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DEVELOPMENT OF REFRACTORY FABRICS

L. E. GATES AND W. E. LENT

8 APRIL 1963

HUGHES

HUGHES AIRCRAFT COMPANY

COMPONENTS AND MATERIALS LABORATORY

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Huntsville, Alabama

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COMPONENTS AND MATERIALS LABORATORY
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ABSTRACT

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The compositions and fiber forming characteristics of 25 refractory glass materials are described. The construction and operating details are given for a vertical arc-fiberizing apparatus developed for rapid fiber formation of refractory glasses. The construction and operating procedure of a new high temperature glass melting furnace for fiberizing are described. The new furnace incorporates a combination of oxygen-acetylene burners and a high-current immersed electrode system to achieve glass temperatures in excess of 1850°C. Evaluations of single fiber properties and the properties of fiber-resin composites are given. The problems of carding brittle fibers are discussed. A testing machine was developed for evaluating high temperature fabrics under dynamic loading and heating conditions. This machine was designed for simulating a flexible heat shield environment and was used to compare the performances of 23 commercial inorganic fabrics.

AUTHOR

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I. SUMMARY

In the course of this work, 151 refractory glass compositions were studied for fiber forming characteristics. The selection of compositions for evaluation was based on contemporary theories of the glass network forming characteristics of various compounds. Acceptable fiber yields were attained with 25 glasses having fusion points exceeding 1450° C, three of which had fusion points greater than 1800° C. Acceptable fibers were produced from the following types of materials: silica-spinel (magnesium aluminate), silica-spinel-zirconia, silica-zirconia, silica-zinc spinel, aluminum phosphate glasses and fluoride glasses. Compositions which did not produce acceptable fibers were high zirconia materials, barium spinels, and calcium aluminate.

A vertical arc-fiberizing apparatus was developed with a capability for fiberizing very different refractory glasses having wide ranges of properties. Although the apparatus was originally designed as a laboratory research tool for the evaluation of many compositions daily, up to 1/4 pound of fibers of a single composition could be produced in an 8-hour day. Fibers up to 6-1/2 feet long were produced with the apparatus.

To increase fiber output, studies were conducted on various glass heating techniques and improvements in the annular fiberizing nozzle. These studies led to the development of a refractory glass melting furnace with the fiberizing nozzle mounted below the furnace at the molten glass discharge orifice. The furnace was operated successfully to produce a continuous glass stream for fiberizing. The final design of the furnace incorporated a water-cooled refractory shell for containing the molten glass and a water-cooled welded stainless steel draining plate with an Inconel orifice insert. A combination of oxygen-acetylene burners and tungsten electrodes immersed in the glass was used to achieve glass temperatures exceeding 1850° C. With a new high pressure fiberizing nozzle small quantities of high quality long staple fibers were produced from a glass having a fusion point of 1810° C. The majority of the time spent on the apparatus was used in developing the

water-cooled insulation, the burner placement and the electrode system to achieve the required heat balance for efficient glass melting and glass temperature control at the draining orifice. The furnace as constructed should ultimately produce in each firing about ten pounds of glass in a continuous stream for fiberizing. In this program this was not demonstrated because of clogging in the new high pressure annular fiberizing nozzle apparently caused by turbulence in the nozzle throat. Despite the clogging problem, the new nozzle appeared to be more efficient in forming acceptable fibers than the low pressure nozzle developed earlier.

Improvements in the testing apparatus for single fiber tensile strength increased the precision of tests conducted on nine fibers. The highest mean tensile strength, a value of 295,000 psi, was obtained with R-141 fibers. Treatment of R-74 fibers with anhydrous Linde A-1100 silane finish improved its mean fiber tensile strength by 25 percent. The lapse of time after fiber formation had no measurable effect on tensile strength.

A static heating test conducted with various high melting fibers indicated that R-108 underwent no significant changes in bulk volume or resiliency on exposure to 2750°F (1510°C) in an oxidizing atmosphere.

For fiber-resin composite fabrication, ten fiber materials were selected on the bases of high fiber yield, fusion temperature, and type of composition. Fiberfrax, a commercial ceramic fiber, was included for comparison.

A new, more effective method of removing pellets from blown fibers was developed. The depelletized fibers were treated with a silane finish and felted into ten-inch diameter felts prior to resin impregnation. Composites containing 30 percent by weight of CTL 91-LD phenolic resin were molded under high pressure from the impregnated felts and post-cured to achieve optimum properties.

Flexural strength, flexural modulus of elasticity, and punch shear strength tests were conducted on the composite specimens. The highest average flexural strength obtained was 19,958 psi with the R-74 fiber-resin composite. This compares very favorably with the military specification of 13,000 psi flexural strength for randomly oriented fiber

reinforced composites. The highest punch shear strength (11,509 psi) was obtained with the R-89 fiber-resin composite. The effects of anhydrous fiber finishes on composite strength were not clearly indicated.

Plasma arc tests at a heat flux of 550 BTU/ft^2-sec on eight composite materials indicated ablation rates generally equivalent to Fiberglas-Micarta No. 259-2. The composite reinforced with R-99 fibers had an average ablation rate of 0.008 inch per second and appears quite promising on the basis of these tests.

As a result of a survey of several industrial plants and universities the carding operation was ascertained to be the most important and difficult problem in processing ceramic fibers into fabrics. A subcontract on carding techniques for ceramic fibers was issued to the A. French Textile School, Georgia Institute of Technology. A degree of success in forming a web and sliver was obtained with a 1 to 1 mixture of ceramic and asbestos fibers on asbestos carding equipment. Carding studies on conventional carding machinery with raw fibers were not successful; however, a weak web was formed with fibers coated with an organic lubricant. At Hughes Aircraft Company several unconventional fiber handling techniques were explored. Swirling bundles of fibers with air jets showed promise in orienting the fibers into long strands. Also, several feet of strong yarn (tensile strength, 1630 psi) were hand twisted and braided in water without excessive fiber breakage.

A dynamic fabric testing apparatus was developed with which specimens of ceramic fabrics could be subjected to simultaneous dead weight loadings and extreme cyclic transverse displacement of one end in the plane of the fabric, and high radiant heat fluxes normal to one face of the fabric. These parameters simulate the environment of a flexible heat shield on a large rocket booster. By statistical methods a test procedure was established that was utilized for testing specimens of 23 different grades and types of fabrics including asbestos, Fiberfrax, Refrasil, Sil-Temp, Armalon, and Fairprene, with Type 181 E-glass cloth as a comparison standard. Although none of the materials would withstand the actual operating conditions anticipated for the heat shield,

it was noted that two of the asbestos materials showed the best overall performance under load, displacement and heat flux, and that the Refrasil and Sil-Temp showed outstanding heat resistance but poor resistance to high loads. The tests indicate that a strong, high quality asbestos fabric faced with Refrasil or Sil-Temp for heat protection may provide the best combination of materials to resist the heat flux and loads anticipated in the flexible heat shield.

Tests in the above apparatus on the effects on fabric life of reflective inorganic pigments applied to E-glass fabric indicated a 50 percent improvement in lifetime with a magnesia coating. Further work on reflective pigments was deemed very worthwhile.

II. PURPOSE

The purpose of the initial part of this program, entitled "Development of Ceramic Fibers for Reinforcement in Composite Materials," and covering the period 2 October 1960 to 2 December 1961, is as follows: Conduct research directed toward the development of ceramic fibers and ceramic fiber composites for use in space technology. The work shall include but not be limited to the following:

1. Develop refractory glass compositions with characteristics suitable for fiber forming.
2. Develop methods for forming fibers from the above refractory glass compositions.
3. Study the pertinent fiber forming parameters for each promising composition as necessary to achieve reproducibility.
4. Conduct physical, mechanical, and thermal property tests and petrographic analysis on promising fibers.
5. Fabricate fiber-matrix composite test specimens and conduct physical, mechanical, and thermal property tests on these specimens.
6. Furnish specimens to MSFC of promising fibers and composites (organic or inorganic matrix as required) obtained as a result of the above.
7. Develop quantity production methods for fiber forming.
8. Develop inorganic matrix materials compatible with the ceramic fibers; prepare test specimens and conduct property tests on these specimens.
9. Ascertain the feasibility of ceramic fiber-composite materials for use in structures for cryogenic and hyperthermal applications.

The purpose of the final part of this program, entitled "Development of Refractory Fabrics," and covering the period 3 December 1961 to 2 December 1962, is as follows:

This program is directed toward the development of refractory fibers and, ultimately, weaving of these fibers into fabrics which shall be evaluated for potential use as insulating or heat blocking media under mechanical stress in hyperthermal environments.

III. EXPERIMENTAL WORK

A. CERAMIC FIBER COMPOSITIONS

In the course of this contract and a preceding program,* 151 ceramic compositions were studied in a search for refractory glasses that would form fibers. The compositions, fiber characteristics and fusion points of all promising compositions are given in Table 1. The glass fiber fusion temperature was considered a major factor in the selection of new materials for more advanced studies. This property was determined according to the ASTM test No. C24-46 for Pyrometric Cone Equivalent (PCE) of Refractory Materials. Standard PCE cones were prepared with fibers produced from those compositions with the highest relative fiber yields. The use of fibers rather than the raw composition constituents assured that any portion of the constituents volatilized during arc fiberizing would not be present and the fusion temperatures would be those of the actual glasses composing the fibers. In several cases where no fibers were formed but glassy pellets suggested a glass-forming tendency, fusion temperatures of the pellets were determined. The pellets were assumed to represent the true composition of a glass melted in the fiberizing apparatus since it was unlikely that further volatilization of a constituent would occur after a molten droplet was removed from the arc because of the sharp drop in temperature.

There were 25 materials which appeared promising for advanced studies in the arc fiberizing apparatus. All of these materials had acceptable fiber yields of more than one gram per fifty blasts and fusion points exceeding 1450° C. With these materials, a variety of fiber characteristics were manifested such as fiber length and diameter, flexibility and brittleness, melting behavior (surface tension and viscosity), and resistance to devitrification. Several fiber compositions were

*Contract No. DA-04-495-ORD-1723, "Studies on Refractory Fiber Research," Army Ballistic Missile Agency, U.S. Army Ordnance Corps.

Composition Number	Composition		Fiber Yield gm per 50 Blasts	Fiber Fusion Temperature °C	Comments
	Material	Percent			
R45	SiO_2	36.00	6.6	1475	Fine long fibers, low viscosity
	Al_2O_3	48.00			
	MgO	16.00			
R58	SiO_2	25.00	3.0	1495	Extremely fine short fibers
	Al_2O_3	56.00			
	MgO	19.00			
R59	SiO_2	35.50	5.8	1450	Very fine short fibers, high viscosity
	Al_2O_3	30.50			
	MgO	9.50			
	ZrO_2	24.50			
R66	SiO_2	42.75	1.4	1500	2 - 3 inch fine fibers
	Al_2O_3	15.25			
	MgO	4.75			
	ZrO_2	37.25			
R74	SiO_2	50.00	8.6	1450	Fairly fine long fibers, easy to fiberize
	Al_2O_3	22.50			
	MgO	7.50			
	ZrO_2	20.00			
R76	SiO_2	60.00	3.2	1605	1 - 6 inch fine fibers, difficult to fiberize
	Al_2O_3	7.50			
	MgO	2.50			
	ZrO_2	30.00			
R79	SiO_2	20.00	3.0	1550	4 - 6 inch fine fibers
	Al_2O_3	45.00			
	MgO	15.00			
	ZrO_2	20.00			
R86	SiO_2	45.00	1.2	1680	Short fibers, very high viscosity
	ZrO_2	45.00			
	PbO	10.00			
R87	SiO_2	40.00	2.0	1680	Medium fibers, very high viscosity
	ZrO_2	40.00			
	ZnO	20.00			
R89	SiO_2	35.00	1.5	1810	Medium fibers, good quality
	Al_2O_3	32.50			
	ZnO	32.50			
R91	SiO_2	25.00	2.0	1620	2 - 4 inch fine fibers
	Al_2O_3	62.50			
	MgO	12.50			
R99	SiO_2	50.00	7.0	1580	Long flexible fine fibers
	Al_2O_3	27.00			
	MgO	3.00			
	ZrO_2	20.00			
R108	SiO_2	17.90	1.3	>1830	1 - 2 inch very fine fibers
	Al_2O_3	35.70			
	ZnO	35.70			
	Sb_2O_3	10.70			

Table 1. Selected fiber compositions.

Composition Number	Composition		Fiber Yield gm per 50 Blasts	Fiber Fusion Temperature °C	Comments
	Material	Percent			
R110	SiO_2	18.50	1.2	1830	1/2 - 2 inch very fine fibers
	Al_2O_3	37.00			
	ZnO	37.00			
	Sa_2O_3	7.50			
R112	SiO_2	17.90	1.4	1750	1/2 - 1 inch fine fibers
	Al_2O_3	35.70			
	ZnO	35.70			
	CeO_2	10.70			
R113	SiO_2	18.50	1.6	1750	1 - 2 inch fine, uniform fibers
	Al_2O_3	37.00			
	ZnO	37.00			
	Y_2O_3	7.50			
R117	SiO_2	54.50	1.0	1505	1 - 6 inch glassy fibers
	Al_2O_3	6.83			
	MgO	2.27			
	ZrO_2	27.20			
	P_2O_5	9.20			
R119	SiO_2	60.00	3.4	1575	Medium texture fibers, viscosity lower than R-76
	Al_2O_3	10.00			
	ZrO_2	30.00			
R123	SiO_2	58.20	2.2	1575	1 - 3 inch fine textured fibers, pink color
	Al_2O_3	7.28			
	MgO	2.42			
	ZrO_2	29.20			
	Sa_2O_3	2.90			
R132	SiO_2	45.00	1.2	1790	1 - 3 inch fine fibers
	Al_2O_3	40.00			
	P_2O_5	15.00			
R141	Al_2O_3	57.00	1.4	1530	Low viscosity melt, 1 - 2 inch fine fibers
	CaF_2	43.00			
R145	SiO_2	57.70	1.0	1485	Fine short dark gray fibers
	Al_2O_3	7.20			
	MgO	2.40			
	ZrO_2	28.70			
	V_2O_5	4.00			
R147	SiO_2	56.96	1.8	1500	Somewhat viscous melt; short fine fibers
	Al_2O_3	7.08			
	MgO	2.36			
	ZrO_2	28.60			
	CeO_2	5.00			
R149	SiO_2	24.60	2.4	1500	Low viscosity, high surface tension; 2 - 3 inch fibers
	Al_2O_3	55.40			
	CaF_2	20.00			
R150	SiO_2	21.20	2.4	1520	Low viscosity, high surface tension; 2 - 3 inch fibers
	Al_2O_3	43.80			
	CaF_2	10.00			
	ZnO	25.00			

Table 1 (continued).

found to be superior as reinforcements in resin composites (e.g., R-74 and R-89) and others appeared to be more suitable for fabrics (e.g., R-99). Satisfactory fiber forming abilities of the 25 compositions have been demonstrated in the vertical arc fiberizing apparatus. It is considered practical to produce larger quantities of fibers by other fiberizing methods from any of these materials as required for yarn and fabric processing.

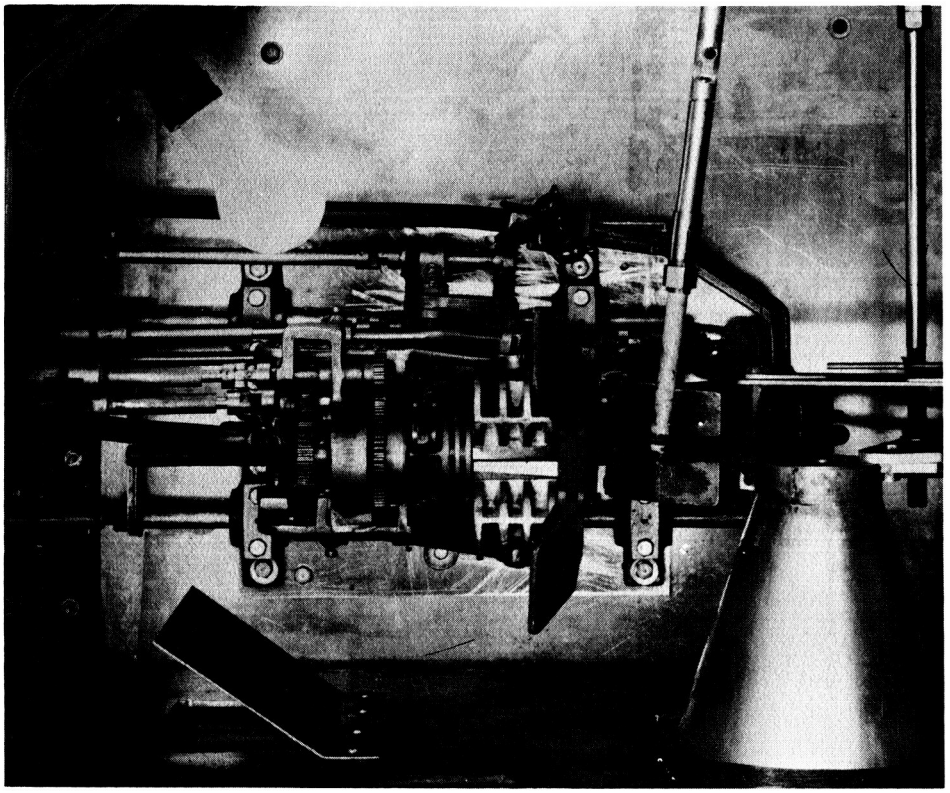
Of the various types of materials studied, the following were capable of producing acceptable refractory fibers: silica-spinel (magnesium aluminate), silica-spinel-zirconia, silica-zirconia, silica-zinc spinel, aluminum phosphate glasses, and fluoride glasses. Compositions which did not produce acceptable fibers were high zirconia materials, barium spinels, and calcium aluminates.

Those materials selected for studies in the refractory glass melting furnace (q.v., below) were R-74, R-99 and R-89, in order of increasing fusion points.

B. FIBER FORMING

Vertical Arc Fiberizing Apparatus

The initial studies on forming refractory fibers were conducted with an arc fiberizing apparatus. In this device, Figure 1, a rod of material was fiberized by melting the tip in a high intensity d-c arc, removing it momentarily to cool, encircling the tip with an annular nozzle, and blasting fibers from the tip with high pressure gas jets. For uniform heating the rod was rotated in the arc by an electric motor. Timers were provided for automatic control of blast delay and blast duration and were adjustable over wide ranges to suit the fiberizing characteristics of different refractory glasses. Studies of various annular nozzle configurations, blast pressures, types of gases, and the operating parameters were effective in improving the fiber quality and yield and in increasing the operating efficiency. Although the apparatus was designed as a research tool for the evaluation of many compositions daily, with a single high yield material up to one-fourth pound of depelletized fibers could be produced in an eight-hour day, Figure 2.



Arc apparatus interior and ceramic rod

Exterior arrangement and controls

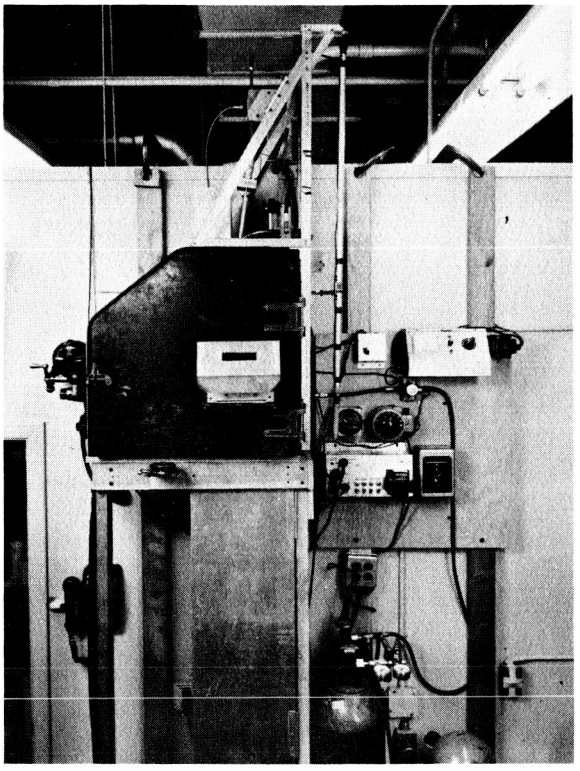


Figure 1. Vertical arc fiberizing apparatus.

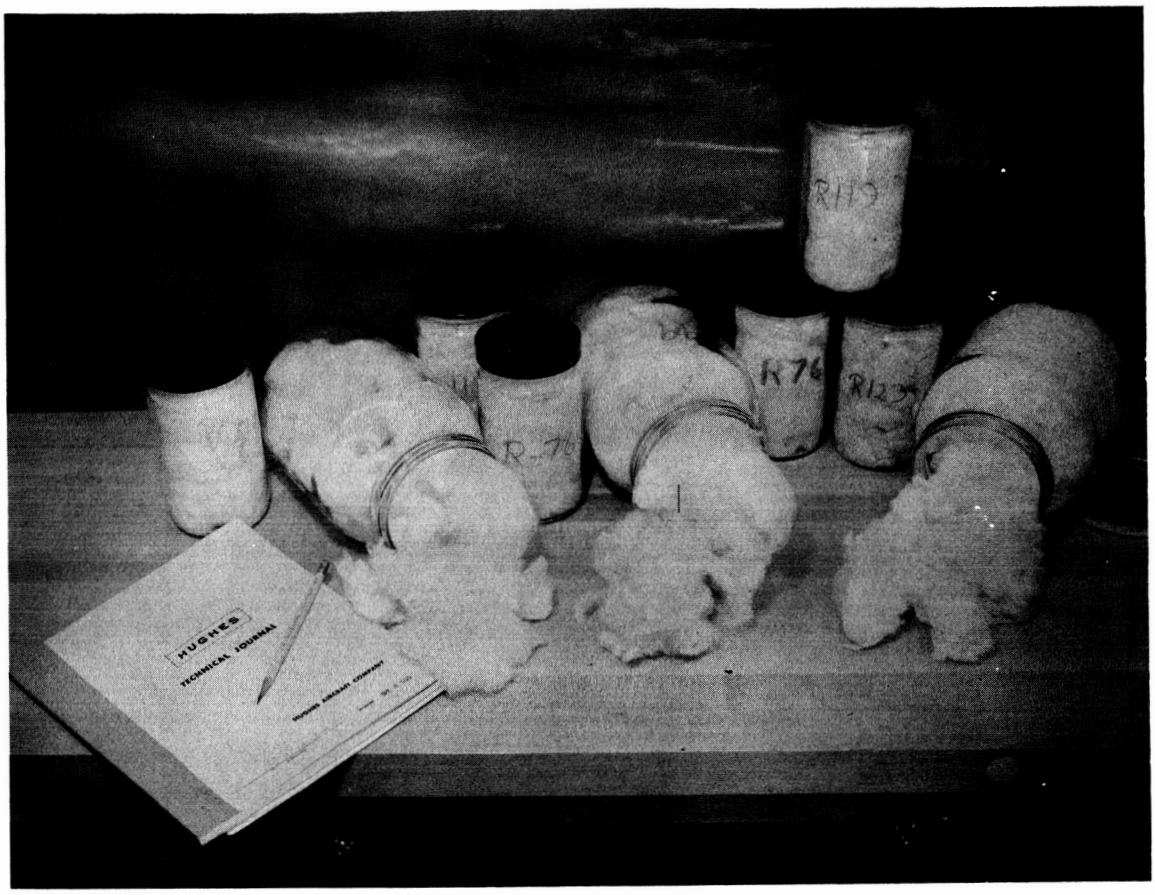


Figure 2. Fibers produced in arc apparatus.

Continuous Fiberizing

The requirements for larger quantities of fibers for fabric studies led to the need for a new apparatus with a higher fiber output. Therefore, a principal objective of fiber formation studies in 1962 consisted of melting a sufficiently large quantity of refractory glass to produce a controlled continuous stream of material for fiberizing. It was considered essential to prevent contamination of the melted glass and highly desirable to eliminate the need for an inert atmosphere. Initial studies of several methods of continuous fiberizing were conducted using a 10-kva Lepel induction power supply to heat a refractory crucible. Initial studies consisted of heating various sizes of graphite rods in a water-cooled induction concentrator to determine the optimum power settings. Attempts were then made to melt various powdered refractory metal borides, carbides, and nitrides and also several glass compositions by self-induction. Even when these materials were heated to 1700° C with a gas flame, there was insufficient electrical conduction to generate internal heat by induction at the operating frequency of this power supply (100 kc).

Two heating methods utilizing a graphite susceptor were then evaluated. The first method consisted of heating the glass in an air atmosphere in refractory crucibles into which close-fitting tubular or rod susceptors were inserted. The crucibles were made of boron nitride or of sintered material of the glass to be evaluated. This method resulted in considerable carbon contamination of the glass from the graphite susceptor, whichever type of susceptor was tried. Also, the boron nitride crucibles were heavily attacked by the molten glass.

The second heating method consisted of suspending a sintered rod of material in a tubular susceptor and heating it by radiation and convection. This method was highly successful, resulting in melting a fairly continuous stream of glass droplets from the tip of the rod as it was lowered into the susceptor. The glass droplets were completely free from carbon contamination. No attempt was made at that time to fiberize the falling droplets. Nevertheless, several inherent problems with this method (including severe oxidation of the susceptor, and an

inability to generate sufficient heat to provide a high volume, continuous stream) led to its abandonment.

There were then two approaches that were considered for achieving a continuous stream of molten glass: (1) a batch process in which a large batch could be melted and completely fiberized before recharging, and (2) a continuous process in which the material could be replenished in a heating chamber at the same rate at which it was removed. The batch process offered the advantage of replacing parts such as eroded nozzles after each run. However, if a continuous process were efficiently designed and engineered, the apparatus could be moderate in size and could be adjusted to provide better control of the fiberizing parameters.

With these considerations in mind, studies of various techniques for melting refractory glasses resulted in a new concept for a melting furnace to achieve the fiberizing temperatures required with maximum efficiency and convenience. Laboratory experiments demonstrated the feasibility of using oxygen-acetylene flames to preheat a glass batch to 1850° C and with immersed tungsten electrodes to continue heating the batch to temperatures far above this temperature. The new method eliminated the need for special crucible and orifice materials since the material to be fiberized acted as its own container. The method also provided precise control of the melt temperature to achieve a uniform, continuous stream for fiberizing by blowing. Although the apparatus was batch operated throughout this program, the design is adaptable for semi-continuous operation.

Development of Fiberizing Apparatus

In the development of the new furnace the first experiments consisted of demonstrating that refractory glasses could be heated with an electric current. An arc was struck between tungsten electrodes located in the bottom of a crucible containing R-74 grain (fusion point, 1450° C). As the electrodes were slowly separated the arc was soon extinguished but current continued to flow through the glass between the electrodes until the crucible was one-third filled with molten glass at a

uniform temperature of 1750° C, with regional heating between electrodes several hundred degrees higher. This experiment was repeated successfully with R-99 and R-89 grain (fusion points 1580° C and 1810° C respectively). There were no difficulties in maintaining a uniform glass melt in this manner and adequate control of the glass temperature was judged feasible with proper design of the electrode system, the crucible and the surrounding insulation materials. The tungsten electrodes showed no significant erosion or oxidation.

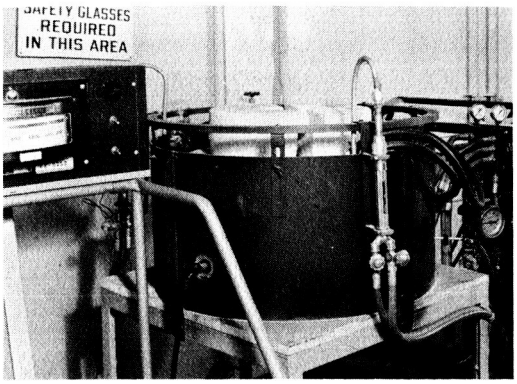
Since a glass must be heated to a temperature at which electrical conduction will occur, a series of experiments was conducted to study techniques of preheating glass batches with oxygen-acetylene flames. The chief considerations were the location of the burners and the configuration and thickness of the insulation required to promote effective heat transfer to the glass. Composition R-99 was selected for these experiments because of its generally satisfactory single fiber characteristics as well as its outstanding fiberizing ability. The experiments consisted of heating and melting R-99 grain in progressively larger hearth-plates fabricated of sintered R-99 grain, with one, two and three burners mounted in castable insulating refractory shells. In each experiment the burner angles, insulation thicknesses and combustion gas outlets were modified as necessary to improve heating efficiency. Although it was soon proved feasible to discharge a continuous stream of molten glass from the hearth, it was apparent that a water-cooled hearth-plate with an appropriately sized orifice was necessary to control the stream diameter and the rate of flow. A water-cooled welded stainless steel hearth-plate with an Inconel orifice insert was ultimately found to work satisfactorily.

When it was found that glass temperatures of more than 1700° C could be consistently achieved with the furnace, an experiment was conducted with an electrical boost system consisting of two tungsten electrodes connected to a 110 volt Variac and inserted through the hearth walls into the glass melting chamber. The heating characteristics from this test gave sufficient information to calculate the power requirements for a three-electrode system.

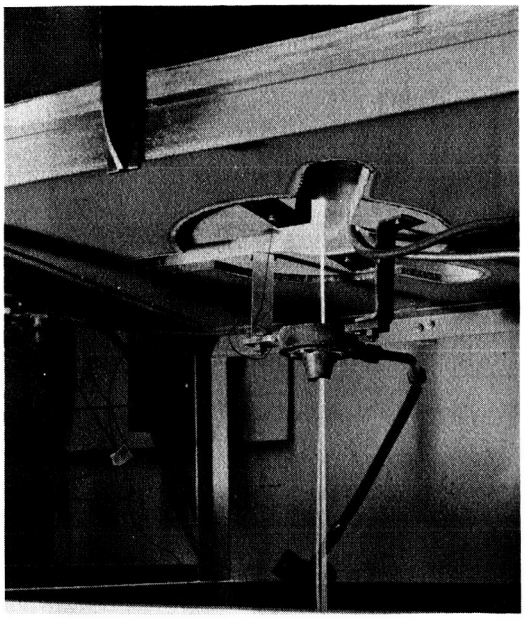
High Temperature Glass Fiberizing

A new water-cooled refractory shell was then fabricated with provisions for three electrodes which were wired to the output from three wye-connected, 240 volt Variacs fed from a 208 volt line, Figure 3. Because subsequent experiments were to be conducted with R-89 (fusion point, 1810° C), the water-cooled shell and the improved water-cooled stainless steel hearthplate were included at this time for protection against the considerably higher fiberizing temperature required. The shell consisted of a one inch thick bubble alumina pot containing 1/4 inch copper tubing coils spaced one inch apart in the outer surface. The Type 416 stainless steel hearthplate was of welded construction with a 5/16 inch cooling water passage encircling an Inconel draining orifice insert with a 1/2 inch diameter orifice. The cooling water lines for both the shell and the hearthplate were fitted with separate needle valves and thermometers for precise temperature regulation.

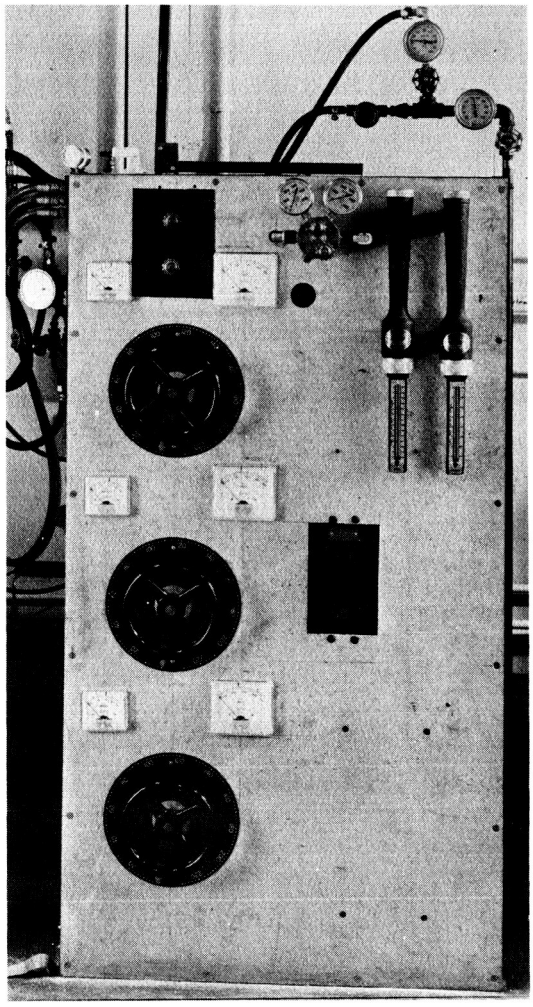
Three firings were then conducted with the chamber charged with R-89 grain and with a new high pressure fiberizing nozzle mounted below the draining orifice. In the first firing the glass began to conduct an electric current at a glass temperature of 1700° C. The furnace temperature was permitted to rise gradually while adjustments were made in the cooling water supply to the shell and hearthplate, in the electrode spacing and in the power settings to the electrodes. The electrode power distribution was easily balanced and controlled by adjusting voltage and current outputs with each Variac. It was found that the heat could be either concentrated over the draining orifice or distributed evenly throughout the melt by inserting or withdrawing the electrodes. The maximum power input to the three electrodes ranged from 20 to 25 kw. In each firing when the glass appeared to have been uniformly melted, a tungsten rod was pushed up from below and quickly withdrawn to open the orifice. A thin glass stream started to flow, but about ten seconds after fiberizing began, the fiberizing nozzle became plugged with glass and the furnace had to be shut down. In an attempt to overcome this in the second firing the nozzle was raised closer to the draining orifice and the blasting pressure was reduced from 900 to 700



Refractory glass melting furnace.



Continuous glass stream flowing before fiberizing.



Furnace control panel.

Figure 3. Gas-electric fiberizing apparatus.

psi. In the second firing again the nozzle became plugged. For the third firing the Inconel insert was replaced with one having a 3/8 inch diameter orifice in an attempt to reduce the glass flow rate. This fiberizing trial also ended in plugging of the nozzle, which is apparently due to the small opening (3/4 inch) in the nozzle, or perhaps to a slightly turbulent back pressure above the nozzle. The results of fiberizing in these three firings are given in Table 2.

No further experiments were conducted after this firing, but a close inspection of the shell, electrodes, hearthplate and burners indicated that there was no significant erosion or other deterioration of any part of the apparatus.

Physical Properties	Firing No. 1	Firing No. 2	Firing No. 3
Fiberizing Time - sec.	10.0	10.0	8.0
Fibers Produced - grams	78.0	20.0	51.0
Average Length - inches	2.1	4.4	2.8
Average Diameter - microns	13.2	16.5	15.9
Usable Fibers (after depelletizing) - grams	6.0	8.0	11.0
Average Length (after depelletizing) - inches	0.8	1.5	1.4
Yield (after depelletizing) - percent	7.8	40.0	22.0

Table 2. Results of fiberizing R-89.

C. FIBER EVALUATION

Fiber Dimensions

The diameters of eight promising fibers developed in the vertical arc-fiberizing apparatus were determined optically with a microscope fitted with a measuring filar eyepiece and an oil immersion objective lens. The diameters of ten randomly picked fibers of each material are as follows:

Fiber	Fiber diameter – microns		
	Minimum	Maximum	Average
R45	14.7	36.7	26.6
R74	12.3	22.0	16.1
R76	17.2	50.4	25.5
R89	12.9	43.4	24.8
R99	7.4	30.2	20.1
R108	6.1	19.6	13.5
R119	13.5	46.6	25.9
R123	17.2	41.0	23.1

Fiber Tensile Strength

Evaluations of the tensile strengths of nine selected fiber materials were conducted with an improved tensile strength apparatus, Figure 4. In this apparatus a convenient method of heating and melting the mounting wax was employed which consisted of small individual heaters operated by a treadle. A linkage mechanism which prevented shattering of the fiber ends after breakage reduced the "mortality" (aborted test results) from over 90 percent to less than five percent and also somewhat reduced the usual spread of test data.

The results of these tests on the nine fiber materials are presented graphically in Figure 5. Ten specimens were tested for each bar in the figure. The composition with the highest mean fiber tensile strength was R-141 with a value of 295,000 psi. However, since the two sigma limits of all materials overlap, statistically there is no significant difference in the strengths of the different fibers.

The effects of non-aqueous glass fiber finishes on strength were also studied in the course of this evaluation. Two groups of freshly made R-74 fibers were treated respectively with one percent Quanta 1025C* in

*Thalco, 6431 Flotilla Street, Los Angeles 22, California.

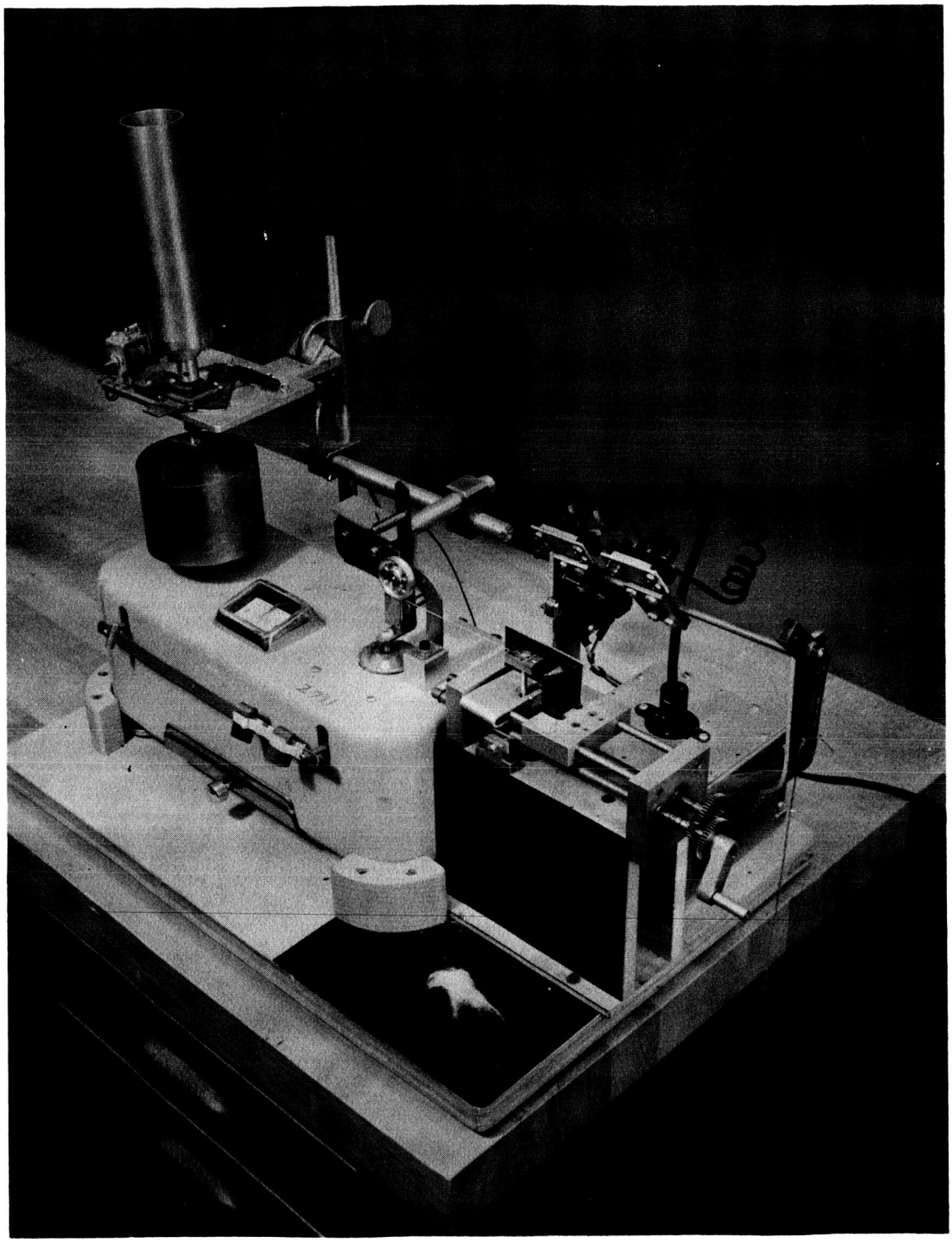


Figure 4. Fiber tensile strength apparatus.

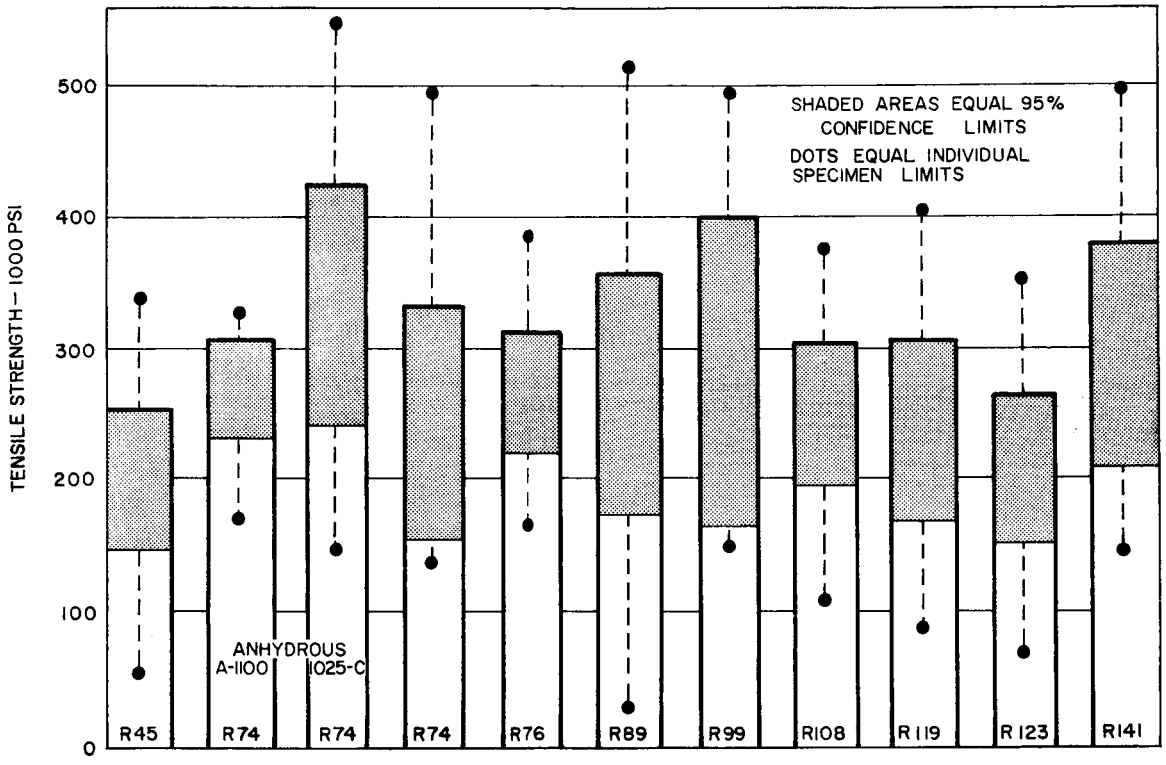


Figure 5. Tensile strengths of selected single fibers.

methyl ethyl ketone and with ten percent Linde A-1100* in anhydrous xylene. After immersion the fibers were washed with water to hydrolyze the finishes as prescribed by their manufacturers. The fibers were then dried and evaluated. The results are also shown in Figure 5. The A-1100 finish improved the mean tensile strength of R-74 by 25 percent. The 1025C finish lowered the tensile strength and increased the scattering of data.

To study the effect of time on the strength of fibers, R-74 fibers from a freshly made batch were tested in various time increments over a twelve-day period. In Figure 6 it is seen that the expected sharp decrease in strength with time is not indicated. Significant differences would be noted, perhaps, with a more precise apparatus.

Static Heat Resistance

To determine the resistance of various bulk fibers to 2750 °F (1510 °C) a static heating test was conducted. With each material to be tested about one-half gram of depelletized fibers was pressed gently into a loose ball about three-fourths inch in diameter and placed on a setter ring, Figure 7, in an oxygen-acetylene fusion furnace. The fibers were heated at the rate of about 500 °C per hour to 1520 °C and the furnace was shut off and permitted to cool normally to room temperature. The results are shown in Figure 7 and are listed in order of decreasing resistance in Table 3. The temperature resistance of Fiberfrax appeared to be better than that of R-89 and R-108. This was unexpected because the fusion point of Fiberfrax was 1740 °C, or 70 °C and 90 °C lower, respectively, than R-89 (1810 °C) and R-108 (>1830 °C). Since the Fiberfrax was placed down inside the ring; apparently it was somewhat sheltered from the same degree of radiant and convective heating to which the other materials were subjected. It is noteworthy that the poorest performer of the refractory fibers tested was zirconia "B" and that "E" glass was completely melted and vaporized.

*Silicones Division, Union Carbide Corporation, 2770 Leonis Blvd., Los Angeles (Vernon) 58, California.

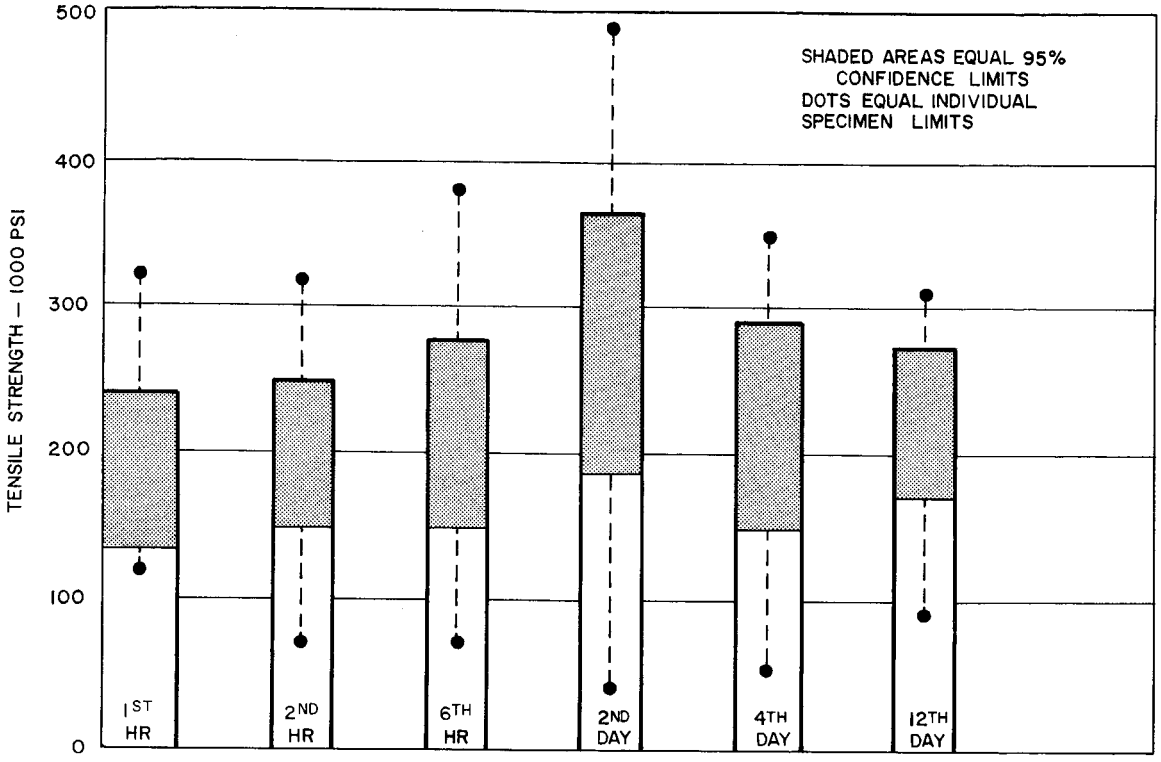
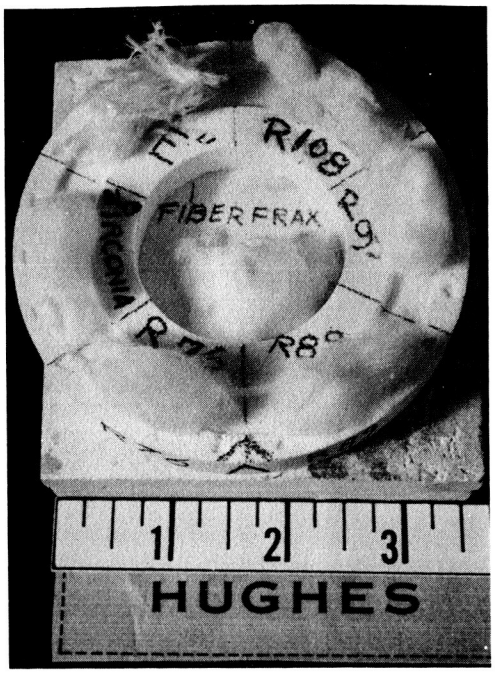
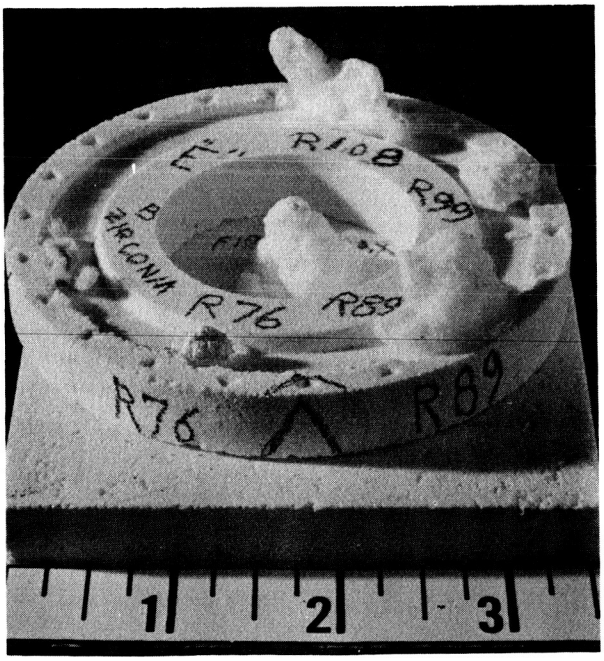


Figure 6. Effect of time on tensile strength of fibers.



Before test



After heating to 1510°C (2750°F)

Figure 7. Static heat resistance of fibers.

Fiber	Shrinkage	Shape Retention	Fusion	Resiliency
Fiberfrax[1]	Some	Good	Slight	Very good
R-108	Some	Good	Slight	Good
R-89	Some	Fair	Slight	Fair
R-99	Excessive	Poor	Excessive	Poor
R-76	Excessive	Poor	Nearly complete	Poor
Zirconia "B"[2]	Extreme	Poor	Nearly complete	Poor
"E" glass	Vaporized	-	-	-

1. The Carborundum Company, Niagara Falls, New York.
2. H. I. Thompson Fiber Glass Co., Inglewood, California.

Table 3. Static heat resistance of fibers.

D. FIBER-RESIN COMPOSITES

Materials Selected for Composites

The selection of the final fiber compositions for volume fiberizing, felting, and composite fabrication was based on three factors – high yield, high fusion temperature, and, where significant, the type of composition, with emphasis placed on yield because of the larger quantities of fibers required for fabrication of composite test specimens. The selected fibers are shown in Table 4, which lists yield, fusion temperature, and composition type. Fiberfrax was included in the composite tests for a comparison of the new fibers with a typical commercial material.

Composite Fabrication

The fibers selected for composite studies were depelletized by swirling small batches in a closed circular chamber with a screen opening in the periphery. The cleaned fibers were felted with a one-gallon Waring Blendor in forty gram batches. The suspension fluid was 3500 cc

of a two percent solution of Linde A-1100 silane finish in water. The blendor was permitted to agitate the suspension for 2-1/2 minutes at a low speed after which the fibers were poured into a ten-inch Buchner filtering funnel connected to a vacuum pump. The fibers settled on the filter paper and the resulting felt was free of pellets, very uniform in texture and thickness, and the fibers were randomly oriented and well intermeshed in the plane of the felt, Figure 8. Two separate batches of R-74 fibers were treated prior to felting with the two non-aqueous glass fiber finishes previously described.

Composition Number	Fiber Yield gm per 50 Blasts	Fusion Temperature Degrees C	Type of Composition
R45	6.6	1475	Magnesia-Spinel plus Silica
R74	8.6	1450	Magnesia-Alumina-Zirconia-Silica
R76	3.2	1605	Magnesia-Alumina-Zirconia-Silica
R86	1.2	1680	Silica-Zirconia plus Flux
R89	1.5	1810	Zinc-Spinel-Silica
R99	7.0	1580	Magnesia-Alumina-Zirconia-Silica
R108	1.3	1830	Zinc-Spinel plus Silica and Flux
R119	3.4	1575	Magnesia-Alumina-Zirconia-Silica
R123	2.2	1575	R76 plus Flux
R141	1.4	1530	Calcia-Alumina-Fluoride (no Silica)

Table 4. Fiber compositions selected for composite specimens.

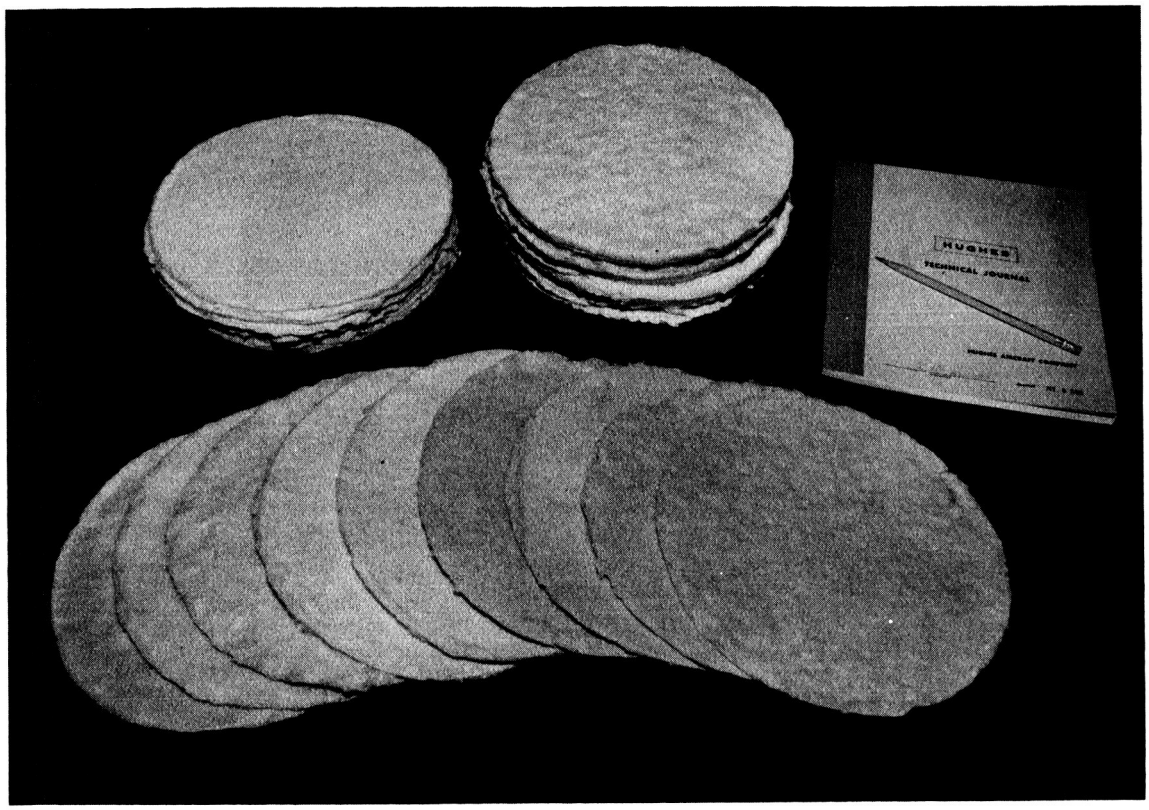


Figure 8. Ceramic fiber felts for composite specimen fabrication.

The ten-inch felts of the selected fibers (treated with the appropriate fiber finish) were impregnated by soaking them in the following resin solution:

Fibers	70 parts by weight
CTL 91-LD resin	43 parts by weight of 70 percent varnish
Acetone	43 parts by weight

The 43 parts by weight of 70 percent varnish is the equivalent of 30 parts by weight of resin solids. The 70-30 fiber-resin ratio was selected because of the improved high temperature performance of composites made with a lower resin content. The impregnated felts were dried for three hours at 90° C, after which two-inch disc and one inch by eight inch bar premold specimens were cut from the felts. The premold specimens were then placed in the appropriate dies and molded as follows:

Die	Die Temperature	Molding Time	Total Pressure
2 inch disc	150° C	60 minutes	4 tons
1 inch by 8 inch bar	150° C	60 minutes	10 tons

The specimens were postcured according to a programmed heating cycle as follows: maintain specimens 18 hours at 275° F, increase temperature gradually in 42 hours to 400° F, maintain four hours at 400° F.

Composite Evaluation

All strength testing was conducted at room temperature with a Baldwin-Tate-Emery Testing Machine. Flexural strength tests were performed according to the ASTM D790-58T test procedure using a test specimen of approximately 0.1 x 0.5 x 4 inches, and a test span of two inches. The modulus of elasticity in flexure was also determined. Punch shear tests were conducted with the 2-inch discs according to the ASTM D732-46 testing procedure.

It was seen that the R-74 composite had the highest average flexural strength, 19,958 psi, and that R-89 had the highest punch shear strength, 11,509 psi. This did not agree with the single fiber tensile strengths

reported for these materials. The effects of anhydrous finishes were also not clearly indicated and did not agree with the single fiber tensile strengths.

Plasma arc tests were conducted on composite specimens under a heat flux of 550 BTU/ft^2-sec. Most specimens had a fairly uniform ablation rate of 0.011 to 0.016 inch per second. The composites containing R-99 fibers had the lowest ablation rate, an average of 0.008 inch per second for two specimens of different thicknesses. The ablation rates of most of these materials agreed with a value of 0.013 inch per second for Fiberglas-Micarta No. 259-2 as reported by the Engineering Experiment Station at Georgia Institute of Technology.

E. FABRIC DEVELOPMENT

Carding Studies

From the results of a survey of several industrial plants and universities early in 1962 the carding operation was ascertained to be the most important and difficult problem in processing ceramic fibers into strong fabrics. The fibers are much too brittle to withstand the severe bending around the wire combs in a conventional carding machine. One producer of ceramic fabrics utilizes an organic fiber carrier web to support the ceramic fibers through the carding, spinning, and weaving operations, but the fabric strength is drastically lowered when the organic material is burned out. The only information obtained from the literature on this problem concerned techniques for carding asbestos, a much more flexible fiber.

For these reasons two approaches for studying the carding problem were undertaken: a small program with the A. French Textile School, Georgia Institute of Technology, to study methods of modifying conventional carding techniques and a parallel program at Hughes Aircraft Company to study new, entirely different carding principles.

Subcontract on Carding Studies

The subcontract issued to the A. French Textile School consisted of two phases: (1) a study of the feasibility of using conventional textile

machinery for processing ceramic fibers into fabrics, and (2) the establishment of criteria based on the characteristics of ceramic fibers in order to develop special machinery to process ceramic fibers into fabrics without the use of organic carrier fibers. The program was completed on 2 December 1962 and the work accomplished is described in the following section as reported by the Project Director, Professor J. W. McCarty of the A. French Textile School.

Final Report, Project No. A-638
"Experimental Carding and Weaving of Ceramic Fibers"
Hughes Aircraft Company P. O. 4-712607-FF31-3
Period – 25 June to 3 December 1962

I. Summary

Experimental carding of treated and untreated Fiberfrax fiber was carried out on existing types of textile carding equipment available in the A. French Textile School. When carding of the ceramic fiber was not found possible with ordinary machinery set up, minor adjustments were made on the equipment such as lowering of speeds and changing of settings. Webs were formed from Fiberfrax fibers, having treatments PF-4, PF-14 and PF-15 (described later), but none were strong enough for continuous gathering into a sliver. A sliver was formed on asbestos carding equipment from a blend of 50 percent untreated Fiberfrax fibers and 50 percent asbestos fibers.

An attempt was made to form the Fiberfrax fibers into paper which could be slit and twisted into a form suitable for use on textile equipment. The papers made were not strong enough to permit twisting.

A brief investigation of the feasibility of electrostatic fiber alignment equipment was made. Alignment was tried, initially using one-half inch conventional glass fiber bundles on an adhesive coated base material. However, the tangled tufts of Fiberfrax ceramic fibers could not be separated well enough to permit these fibers to be propelled through the charged grid of the machine.

II. Objective

The long range objective of this program was to develop techniques of weaving ceramic fibers into fabric (without use of carrier fibers) in such a manner as to retain maximum fiber length and strength. A more immediate and necessary step before the ultimate objective could be met was to form a sliver or roving from the fibers by the use of textile carding machinery or other fiber orienting equipment.

III. Experimental Procedure

The first steps taken involved the processing of the fibers through various commercial types of textile machinery located in the A. French Textile School. The type of machinery used and its description is listed below.

1. REGULAR COTTON CARD with revolving flats.
2. GRANULAR COTTON CARD. This is an ordinary cotton card in which the revolving flats have been replaced with a granular top. The action of this type of top over the cylinder is said to be much less harsh and more gentle to the fibers.
3. ROLLER TOP CARD. This is an ordinary cotton card in which the revolving flats have been replaced by six wire covered rolls – three larger rolls called the worker rolls and three smaller rolls called the stripper rolls.
4. WOOLEN CARD. The ordinary woolen card consists of a battery of three sections – breaker, intermediate and finisher – with a "Bramwell-type" feed at the beginning breaker section and a tape condenser section following the finisher section. Each of the three sections consists of large wire covered cylinders having a series of wire-covered rolls called workers and strippers across the top half of each large cylinder.

The first efforts to card the long staple bulk Fiberfrax fine fibers furnished by Hughes on the equipment described above was almost entirely unsuccessful. Regardless of which card was used

the action was the same for each. Almost all of the fibers were crumbled to dust and dropped under the initial feed roll or the licker-in at the entrance of the machine. A very few fibers which survived the feed roll and licker-in action were so fine that they were deeply embedded in the wire clothing of the cylinder so that the doffer would not remove them for final delivery to the front of the card.

Slight modifications were made on the granular top card. The feed roll was raised until very little pressure was exerted on the fiber as it passed between the feed roll and the feed plate. This made it possible for more of the fibers to reach the licker-in but the action of the licker-in was such that no more fibers passed the licker-in than had in previous tests. A variable speed drive was attached to the licker-in and the speed reduced until it was only one-fourth the normal speed. This speed reduction had no further effect on the carding of the fiber since the bulk of the fiber never reached the licker-in.

A portion of the Fiberfrax fiber was run on a production-line asbestos card at a United States Rubber Company mill. As in all previous attempts on other cards, the fiber could not be carded by itself, but a low strength sliver was produced from a mixture of approximately 50 weight percent Fiberfrax and 50 weight percent of Triple A grade asbestos. It was generally agreed by the personnel from Georgia Tech.and the U. S. Rubber Company that if time were available for proper adjustments to the spacing and speed of the components of an asbestos card that a much improved Fiberfrax-asbestos sliver could be produced.

Five boxes of fibers treated with various organic lubricants and finishes were received from Hughes Aircraft Company and were designated by the numbers: PF-4, PF-11, PF-13, PF-14 and PF-15. A description as to the treatment each of the above samples received is given in a later section. Only a limited amount of time was available to work with them because they were received late in the program. A portion of each of the five samples

was run on the carding equipment available at Georgia Tech.with the best results being obtained on the Granular Cotton Card. On this card, a small amount of samples PF-4, PF-14 and PF-15 passed through the card and was stripped from the doffer by the doffer comb. Only a very small amount of samples PF-4 and PF-15 was discharged at the front of the card – probably less than one percent of the fiber entering the card. However, these two samples had little drop-out under the feed roll or the licker-in due to their crushing action. The majority of the fibers were trapped in the clothing of the main cylinder and the doffer rolls. In contrast, sample PF-14 had much more drop-out under the feed rolls and licker-in, probably approaching 25 to 50 percent of the fiber fed into the card, but had less fiber trapped in the clothing on the main cylinder and doffer rolls. Samples PF-11 and PF-13 behaved in much the same manner as the untreated fibers studied earlier in the contract – that is, the majority of the fibers dropped out at the feed roll and those passing through were trapped in the clothing of the main cylinder.

For the three samples, of which a portion of the material passed through the card, the material was discharged sporadically and had the appearance of downy, duck feathers. In no case was a continuous web formed.

The granular cotton card was readjusted so that all settings were as close as possible. For samples PF-4, PF-14 and PF-15, the percentage of material discharged at the front of the card was increased after the adjustments were made, but the discharge was still not in the form of a web. The doffer and the main cylinder still tended to "load-up" with fine fibers. By touching the doffer roll with a hand card just ahead of the doffer comb a web of fibers could be started and would continue until the doffer comb had made a complete revolution, thus stripping the fibers from the clothing. Attempts were made to pass the short lengths of web formed in this manner through the trumpet and calender rolls but in most

cases the sliver would break almost as soon as it started to form. The web as formed would take very little twisting.

The experience with these three treated fiber samples is encouraging. Additional work should be directed to the addition of the proper rolls to help with the transferring of fibers from the main cylinder to the doffer and some aid to the doffer combs in removing the web from the doffer.

Early in the contract it was observed that standard textile process equipment was not practical with present inorganic fiber technology. A different line of attack was suggested. In recent years a great quantity of paper has been twisted and woven into products on regular textile processing equipment. Since this method does not involve the use of fluted feed rolls as is the case with most textile equipment, it was thought that this technique might be ideal for this application.

Accordingly, arrangements were made with the Herty Foundation of Savannah, Georgia, to try to convert this fiber to a sheet form suitable for slitting and twisting into a paper type of yarn. This Foundation is a state-owned corporation charged with the coordination of science and manufacturing of cellulose products. Specifically this Foundation has done a great deal of fine work in the conversion of pine wood-pulp into papers of various kinds and grades.

The Herty Foundation prepared eleven small hand sheets from the Fiberfrax (long staple, fine fiber) furnished by Hughes Aircraft Company. These sheets were compounded as follows:

1. 100% Fiberfrax without binder.
2. 90% Fiberfrax and 10% National Resyn X-Link 2933 (A vinyl-acrylic copolymer)
3. 90% Fiberfrax and 10% Chemigum 235 (a butadiene-acrylonitrile copolymer)
4. 90% Fiberfrax and 10% Bleached Sulfite Fiber without binder.

5. 75% Fiberfrax and 25% duPont HS1 (a modified cellulose material)
6. 90% Fiberfrax and 10% Chemigum 520 (a butadiene-acrylonitrile copolymer)
7. 90% Fiberfrax and 10% Hycar 2600-30 (a latex acrylic polymer)
8. 80% Fiberfrax, 10% National Resyn X-Link 2833 (see 2 above) and 10% Chemigum 520 (see 6 above)
9. 90% Fiberfrax and 10% Hycar Latex 1571 (a carboxy-modified butadieneacrylonitrile)
10. 90% Fiberfrax and 10% Hycar Latex 2671 (an acrylic emulsion)
11. 100% Fiberfrax (using the long staple medium fiber)

In addition, a twelfth sheet was produced using 50% Fiberfrax, fine fiber and 50% Kaowool.

Quoted below are the comments of the Laboratory Supervisor at the Herty Foundation regarding the results of these experiments:

"This fiber produced a very weak sheet when prepared without the use of internal binders. We found that this fiber could not tolerate the shearing action produced in conventional woodpulp dispersing equipment. The average fiber length was shortened somewhat in the dispersing stage prior to forming the sheets.

Several handsheets were prepared incorporating binders with the fiber. None of the binders used added appreciable strength to the finished sheet. This, of course, does not rule out the possibility that an acceptable sheet can be formed from this fiber. There are many techniques and additives which are yet to be tried in this program."

Our inspection of these sheets confirm the above observations. None of the sheets contain fiber having sufficient length and strength to enable a slitting and twisting process to be performed.

Another method for orienting fibers was investigated – electrostatic flocking. In this process an adhesive is applied to a backing and the backing and adhesive passed through an

electrostatic field by placing the backing on a metal plate connected to the negative terminal of a high voltage power supply. The fibers are in a container distributed evenly over a grid electrode where they receive a positive charge. These positively charged fibers, upon passing through the grid, are projected toward the negative pole and impinge vertically on the adhesive.

This process was first successfully tried with 1/2 inch long bundles of Fiberglas normally used for reinforcement of plastics. A Dekor laboratory flocking machine with 20,000 volt potential, and a hand held plexiglass fiber container with an opening covered by a hardware cloth grid, was used. Fibers were aligned on an adhesive coated backing by shaking the fiber container, allowing the fibers to fall through the positively charged grid onto the backing material which had been placed on the negatively charged plate. It was found, as recommended by the manufacturer, that the fibers worked best in a relative humidity of 60 to 80 percent.

While the 1/2 inch Fiberglas bundles responded well to this technique, efforts to align the Fiberfrax fibers were not as successful. Even after chopping the Fiberfrax fibers to short lengths the tendency was for these fibers to cling together at random angles thus clogging the grid and keeping them from being propelled to the negative plate.

Future Work

Future work to produce a sliver, a yarn and ultimately a cloth from ceramic fibers of the Fiberfrax type should be directed toward a more basic study of the bending behavior of both individual fibers and bundles of fibers and the effects of coating treatments on these fibers. The fact that treated Fiberfrax fibers came through the card not completely broken up while the untreated Fiberfrax fibers were crumbled into powder is encouraging. The sliver produced on the asbestos card when the fiber was blended with asbestos fiber was also encouraging.

A more thorough and detailed study of the carding of treated fiber is recommended, involving the use of a sample card whereby the rolls as well as the size of the card clothing can be readily changed. At the same time such a study is being carried out, a further study to improve the characteristics of staple ceramic fibers, including composition forming equipment and treatment of the freshly formed fibers, should be included.

Fiber Lubricants and Finishes (Hughes Aircraft Company)

A series of seventeen organic and inorganic materials was formulated, Table 5, for coating the raw fibers to promote lubrication during carding. Small bundles of Fiberfrax were dipped in each solution, dried and evaluated for their abilities to allow hand twisting with minimum breakage. From these simple tests five of the formulations were selected for treating twenty-pound quantities of Fiberfrax fine staple fibers which were sent to the A. French Textile School for carding studies as described in the previous section.

Carding Studies at Hughes Aircraft Company

All of the more or less unconventional mechanical carding methods tried in this program broke the brittle fibers into lengths too short for spinning. For this reason work was concentrated on utilizing fluid media to manipulate and orient the fibers. Semi-orientation of fine Fiberfrax was achieved with a closed circular chamber in which a measured quantity of fibers was swirled with air jets for several minutes. Under optimum conditions of fiber quantity, air pressure and time, a definite "stringing" of the fibers into crude strands was achieved. The strands could be twisted by hand into coarse roving much more easily than before the swirling operation. The orienting methods using air (such as combing with air jets) were not successful.

Hand manipulation was even more promising with water as the fluid medium. Fiber strands immersed in water were easily joined and twisted into roving. The water seemed to help lubricate the fibers and cause them to remain in place as the roving was formed. A three-foot

Material Grams	PF-1	PF-2	PF-3	PF-4	PF-5	PF-6	PF-7	PF-8	PF-9	PF-10	PF-11	PF-12	PF-13	PF-14	PF-15	PF-16	PF-17
Distilled Water	7954	7960	4000	7200	7466	7840	7964	7920	7970	7936	7931	7920	7937	7680	7982	7997	7997
Linde A-1100[1]*	46								15	32	23	40	18				
Carbowax 4000[2]		40															
Keltex[3]			0.44														
$PbCl_2$ Glycerine				400 400													
Ethyl Silicate[4]					400												
Nalcoag #1030[5]						160			15								
Colloidal[6] Alumina							36			32							
Colloidal[7] Zirconia								80									
Boron Nitride[8] (200M)											46						
Maccaloid[9]												40					
Hydrochloric Acid					134												
Lubricool Mica[10]													18				
Plastilube[11] Mold Soap														320			
Wetting Agent[12] (X-100)													9				
Karo Syrup (Clear)															18		
Wonder Gum[13] High Viscosity																2.6	
Glamorene[14] Starch																	2.6

*For sources of materials see Table 6.

Table 5. Fiber lubricants and finishes.

1. Linde Air Products Co., 30 E. 42nd St., New York 17, New York.
2. Carbide and Carbon Chemicals Co., New York, New York.
3. Kelco Co., San Diego California.
4. Union Carbide Chemical Co., 30 E. 42nd St., New York 17, New York.
5. Nalco Chemicals Co., 216 W. 66th Place, Chicago, Illinois.
6. "Baymal" – E. I. DuPont de Nemours & Co., Industrial & Biochemicals Dept., Wilmington, Delaware.
7. E. I. DuPont de Nemours & Co., Grasselli Chemicals Dept., Wilmington, Delaware.
8. Carborundum Co., Niagara Falls, New York.
9. The Inerto Co., San Francisco, California.
10. More Power Lubricool Co., 21333 Woodward Ave., Ferndale 20, Michigan.
11. L. H. Butcher Co., 3628 E. Olympic Blvd., Los Angeles, California.
12. Wholesale Supply Co., 1047 N. Wilcox, Los Angeles, California.
13. W. Kent Perkins Processing Specialties, 63 E. Lake St., Chicago, Illinois.
14. A commercial spray starch – shopping markets.
15. E. I. DuPont de Nemours & Co., Industrial & Biochemicals Dept., Wilmington, Delaware.
16. "Mallinkrodt" – Reagent Grade
17. S. Paul Ward Co., 601 Mission, South Pasadena, California.
18. Glasrock Corp., 1101 Glidden St., Atlanta 18, Georgia.

Table 6. Raw material sources.

length of yarn twisted from the wet roving had a dry tensile strength of 150 psi. Another yarn braided wet from three roving strands had an average dry tensile strength of 1630 psi. Although attempts to mechanize these techniques did not prove successful, the possibilities of carding fibers and forming yarn under water or other liquids are highly promising.

Reflective Coatings

Initially, it was anticipated that inorganic reflective coatings applied to ceramic or glass fabrics might improve their resistance to radiant heat. The materials listed in Table 7 were prepared by ball milling four hours with distilled water to obtain a slip of spraying consistency. These materials were applied to Type 181 E-glass cloth and tested as described in the following section.

Raw Materials – Weight Percent	Coating Numbers HPC-1	HPC-2	HPC-3	HPC-4	HPC-5	HPC-6
Magnesia (dense)	100					
duPont titania[15] R901		100				
Calcium carbonate[16]			100			
Baymal colloidal alumina[6]				100		
Calcined Plasmo clay[17]					100	
Fused silica flour[18]						100
Binders:						
Maccaloid[9]	2	2	2	2	2	2
Yellow dextrine[11]	1	1	1	1	1	1

*For sources of materials see Table 6.

Table 7. Inorganic reflective coatings for fabrics.

F. FABRIC TESTING

A study was conducted of the performance of glass, asbestos, Fiberfrax and Refrasil fabrics under simulated environmental conditions for a flexible heat shield on a large booster rocket. A testing apparatus was constructed to subject fabric specimens simultaneously to specified dead weight loadings, high radiant heat fluxes, and extreme cyclic transverse displacement of one end in the plane of the fabric. A statistical

analysis was employed in the comparison of the ceramic and asbestos fabrics with Type 181 E-glass fabric as a standard.

Fabric Testing Apparatus

The framework of the fabric testing apparatus was constructed of 3/8 inch plate steel for ruggedness, Figure 9. Smooth-jawed stainless steel fabric clamps were attached to the framework and to a movable displacement arm which was in turn attached to a double parallelogram linkage to maintain parallelism between the jaws at all times. The displacement arm was connected to the main drive shaft with an eccentric so that the double amplitude transverse displacement could be adjusted from zero to two inches. The main drive shaft was driven by a gear motor through stock gear sets to produce displacement frequencies of one to five cps. The lower jaw was connected through the linkage and a lever arm to an adjustable spring scale for loading the fabric specimen under tension from zero to 60 pounds per lineal inch. The fabric specimen size was three by six inches. The radiant energy source consisted of a set of five G. E. tubular quartz lamps mounted in a Marquardt Corporation ceramic reflector. During a test the reflector was located such that the fabric was 7/8 inch from the front three tubular lamps. A 240 volt Variac was used to adjust the heat flux on the fabric specimen from zero to 83.5 BTU/ft^2-sec. A central power control unit was utilized for stopping the motor, a cycle counter, and the radiant lamps immediately upon failure of a fabric specimen, and for monitoring the power input to the lamps during a test.

Fabric Specimens

The various grades of the materials tested were those suggested by the respective manufacturers for the intended application. All of the samples were supplied free of charge. The general properties of the materials are given in Table 8.

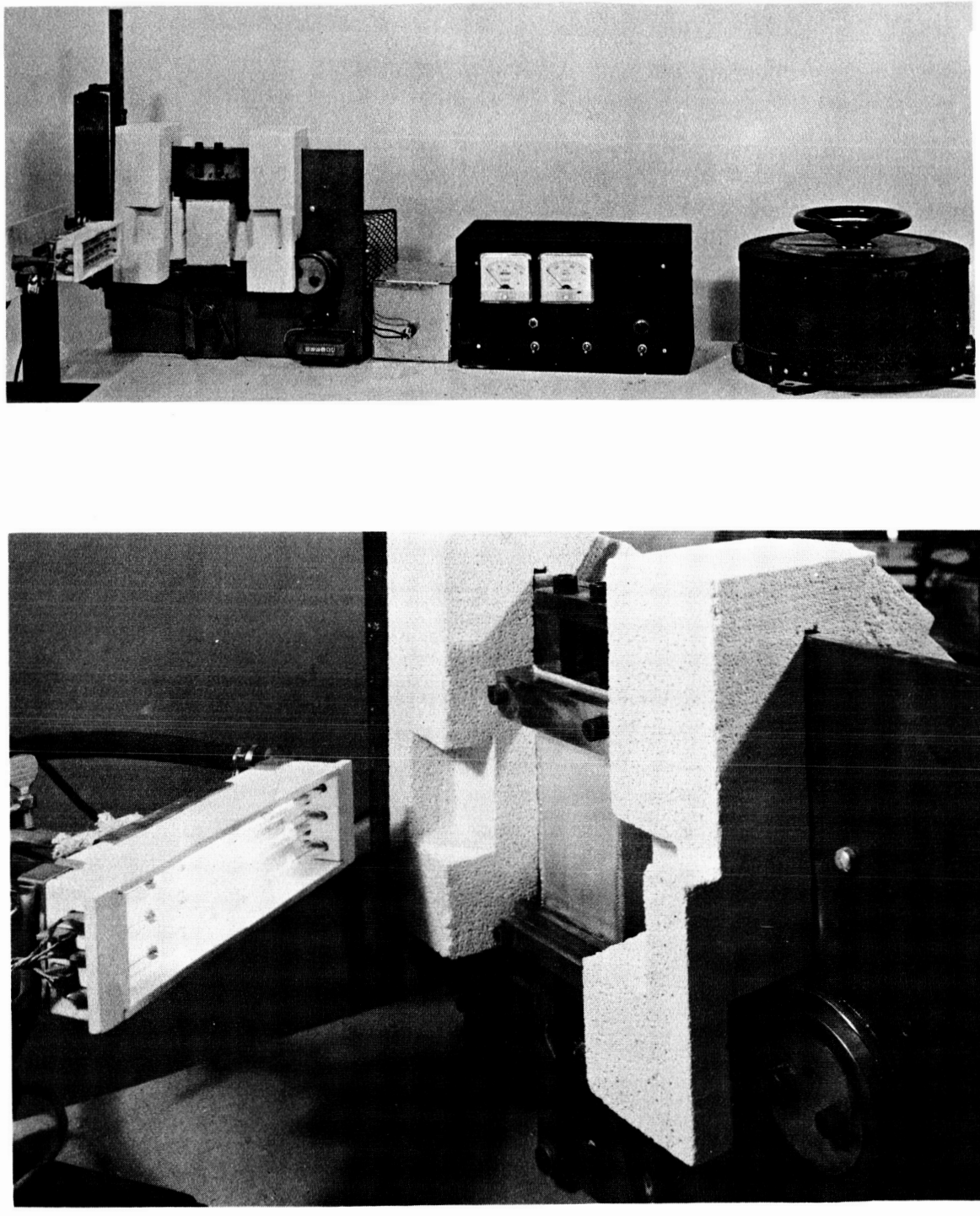


Figure 9. Fabric testing apparatus.

Material	Number	Weave	Yarn Insert	Carrier	Warp and Fill Yarn Reinforcement	Thickness Inch	Comments
Asbestos[1]	12T370	Twill		Cotton		0.085	99-100% asbestos fibers, Grade AAAA
	10P550	Plain		Cotton			95% asbestos fibers, Grade AAA
	24P098	Plain		Cotton			90% asbestos fibers, Grade AA
	10M351	Plain	Inconel	Cotton		0.78	99-100% asbestos fibers, Grade AAAA
Fiberfrax[2]	GL-144T	Twill	Nickel-chrome	Cotton		0.086	Heat treated
	L-144T	Twill	Nickel-chrome	Cotton		0.086	Heat treated
	GL-136	Twill	Stainless steel	Cotton	Glass	0.065	
	L-136	Twill	Stainless steel	Cotton	Glass	0.065	
	TR-122B	Twill	Stainless steel	Cotton	Glass in warp only	0.065	
	P-135	Twill	Stainless steel	Cotton	Glass	0.065	
	L-126	Twill	Glass	Cotton		0.065	
	GL-126	Twill	Glass	Cotton		0.065	
Refrasil[3]	C100-48	Harness					
	C100-96	Knit					
	SS-1451	Twill					
Armalon[4]	414-141						Teflon coated glass fabric
Sil-Temp[5]	85						
	5B						
E-Glass	Type 181						
Fairprene[4]							Laminated glass cloth-neoprene rubber fabric

Sources of Materials:
1. Raybestos-Manhattan Co., North Charleston, S. C.
2. Carborundum Co., Niagara Falls, N. Y.
3. H. I. Thompson Fiberglas Corp.
4. E. I. duPont de Nemours & Co. (Inc.), Wilmington, Delaware
5. Haveg Industries, Inc., Wilmington, Delaware.

Table 8. Description of fabrics tested.

Fabric Testing Procedure and Analyses

The first operation in conducting the fabric tests consisted of calibrating the apparatus by determining the relative importance of the controllable stress factors: load, displacement amplitude and frequency, and thermal flux. Type 181 E-glass cloth was then selected as a standard material for making comparisons between all materials. Finally, the fabric tests and analyses of fabric performance under standard conditions were conducted.

Some preliminary experiments with Type 181 E-glass cloth indicated that there were discrepancies in the resistance of the material to radiant heating depending upon which side of the specimen was exposed. A visual difference in both sides was detected and a series of tests was conducted to ascertain which side, if exposed, would produce the least error.

On one side of the cloth the weave resulted in a pattern in which two sets of intersecting parallel lines appeared. Each set intersected the other set and the horizontal planes of the radiant lamp axes at acute angles. This side was designated side "A." On the reverse side "B" the intersecting angles were obtuse.

Ten specimens of the cloth were prepared for testing. A random combination of the ten digits was used to decide which side of each strip would be exposed to the radiant heat. The test conditions were a static load of 60 pounds per lineal inch and a thermal flux of 60 BTU/ft^2-sec. The time to failure was measured to the nearest second and expressed in fractions of a minute. The test was repeated on another day using a different random combination. An analysis of the results indicated that the test error of side "B" is considerably larger than that of side "A." Therefore side "A" was exposed in all subsequent comparison tests. These results are given in Figure 10.

Since it was not obvious how the three mechanical stress factors should be ranked as to severity of effects, this aspect was then investigated with the E-glass cloth. A factorial experimental design was used incorporating the factor levels:

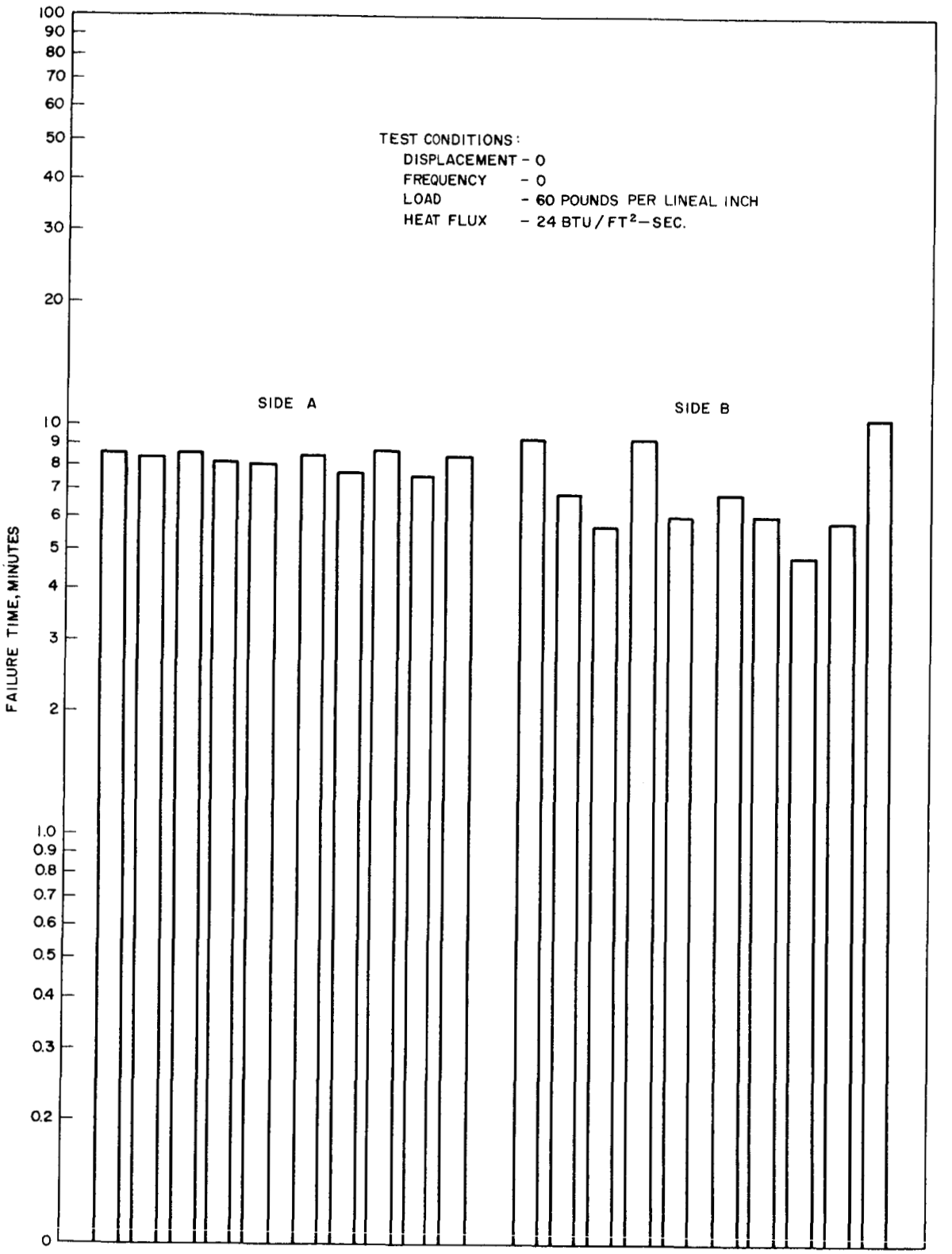


Figure 10. Differences in sides of Type 181 E-glass cloth.

Displacement	0.5 and 1.0 inch
Frequency	1 and 2 cps
Load	10 and 20 pounds per lineal inch

The heat flux was maintained at a constant 10 BTU/ft^2-sec. In order to assure that no heat was reflected from the fabric surface, a 1/2 inch wide stripe of Aquadag colloidal graphite was applied across the center of each specimen, parallel to the lamp axes. The tests were conducted in random order with time to failure as the response and repeated as a replicate. An analysis of the results indicated that the effects in decreasing order of magnitude gave the sequence: load, displacement and frequency. For example, a ten pound decrease in load was much more effective in increasing life span than the 1/2 inch decrease in displacement. Also, the latter was more effective than doubling the frequency. These results are shown in Figure 11.

A similar study of thermal stressing was conducted using the established mechanical stress conditions: load - 10 pounds per lineal inch, displacement - 0.5 inch, and frequency - 2 cps. The thermal fluxes were 5, 10, 15 and 40 BTU/ft^2-sec. Analysis of the test data revealed a slight decrease in time to failure between 5 and 10 BTU/ft^2-sec., but a sharp decrease (two orders of magnitude) in time to failure between 10 and 15 BTU/ft^2-sec. An interpretation of this is that at the lower heat flux the heating apparently reaches a steady state equilibrium with radiative and convective cooling in which the fabric temperature is well below the softening point of the glass. With the higher heat flux transient heating prevails throughout the test and the fabric temperature rapidly rises above the softening point such that failure occurs chiefly by viscous flow rather than by mechanical abrasion. The time to failure at 40 BTU/ft^2-sec. was too short to measure with the colloidal graphite applied to the specimen. However, without the graphite the E-glass cloth survived for a measurable time period apparently because of its high reflectance (and possibly a degree of transmittance).

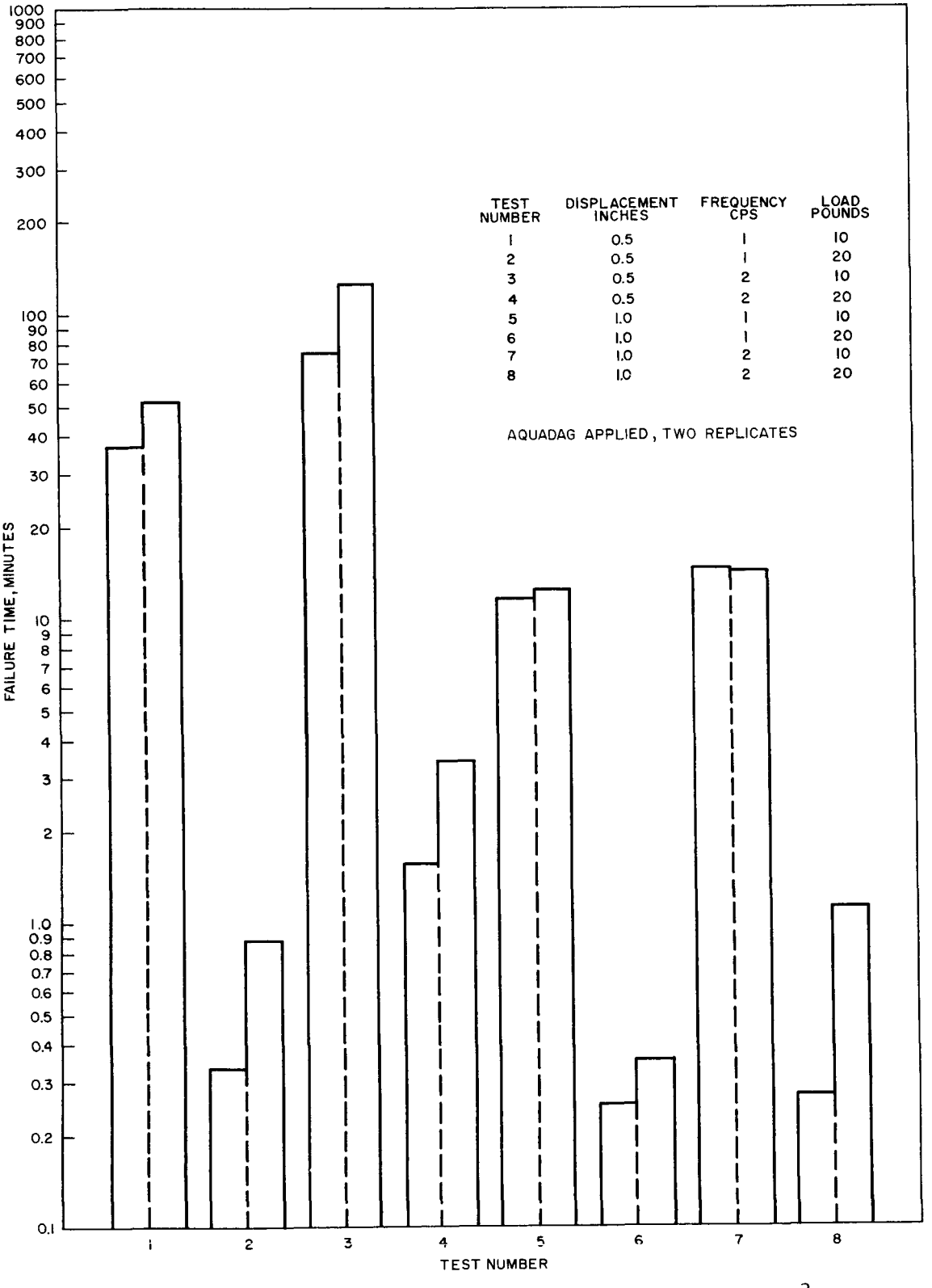


Figure 11. Mechanical stressing of E-glass at 10 BTU/ft^2 -sec.

From the foregoing studies the factor levels were established for initial tests on ceramic fabrics. It must be pointed out that these conditions did not represent the most severe environment specified, but rather they provided an initial standard set of conditions which presumably would allow the majority of the fabric specimens to survive for a measurable period of time so that the materials could be ranked and retested under increasingly severe conditions. It may be stated without qualification that none of the materials submitted would survive the severest environmental conditions anticipated for the flexible heat shield.

All the materials described in Table 8 were tested under the specified initial conditions; the numerical results (time to failure) are shown in Table 9, Column 1, and are compared graphically in Figure 12. A test cut-off point of ten minutes was arbitrarily selected to preserve the life of the infrared bulbs since the anticipated operating time of the flexible heat shield is two minutes. It is seen that four materials survived this ten minute period - three of the asbestos fabrics and one Refrasil fabric. The heat flux was the determining factor in the failure of E-glass, Armalon and Fairprene, whereas the load and displacement limited the life of the asbestos, Refrasil and Sil-Temp.

The fabrics were then subjected to tests under more severe loading and displacement but with reduced heat flux as shown with results in Table 9, Columns 2 and 3, and in Figures 13 and 14. It is again seen that the asbestos fabrics show somewhat more resistance to the loading and heating conditions than the other materials tested.

On comparing the results of all three series of tests it is apparent that the Grade AAAA asbestos materials show the best overall performance. Of the Fiberfrax fabrics, the GL-144T material was more resistant to both load and heat. The Refrasil and Sil-Temp fabrics showed outstanding heat resistance but poor performance under high loads. The results with Fairprene may be somewhat misleading since in both of the tests to which it was subjected the rear face (opposite the exposed glass fabric) rapidly ignited and failed

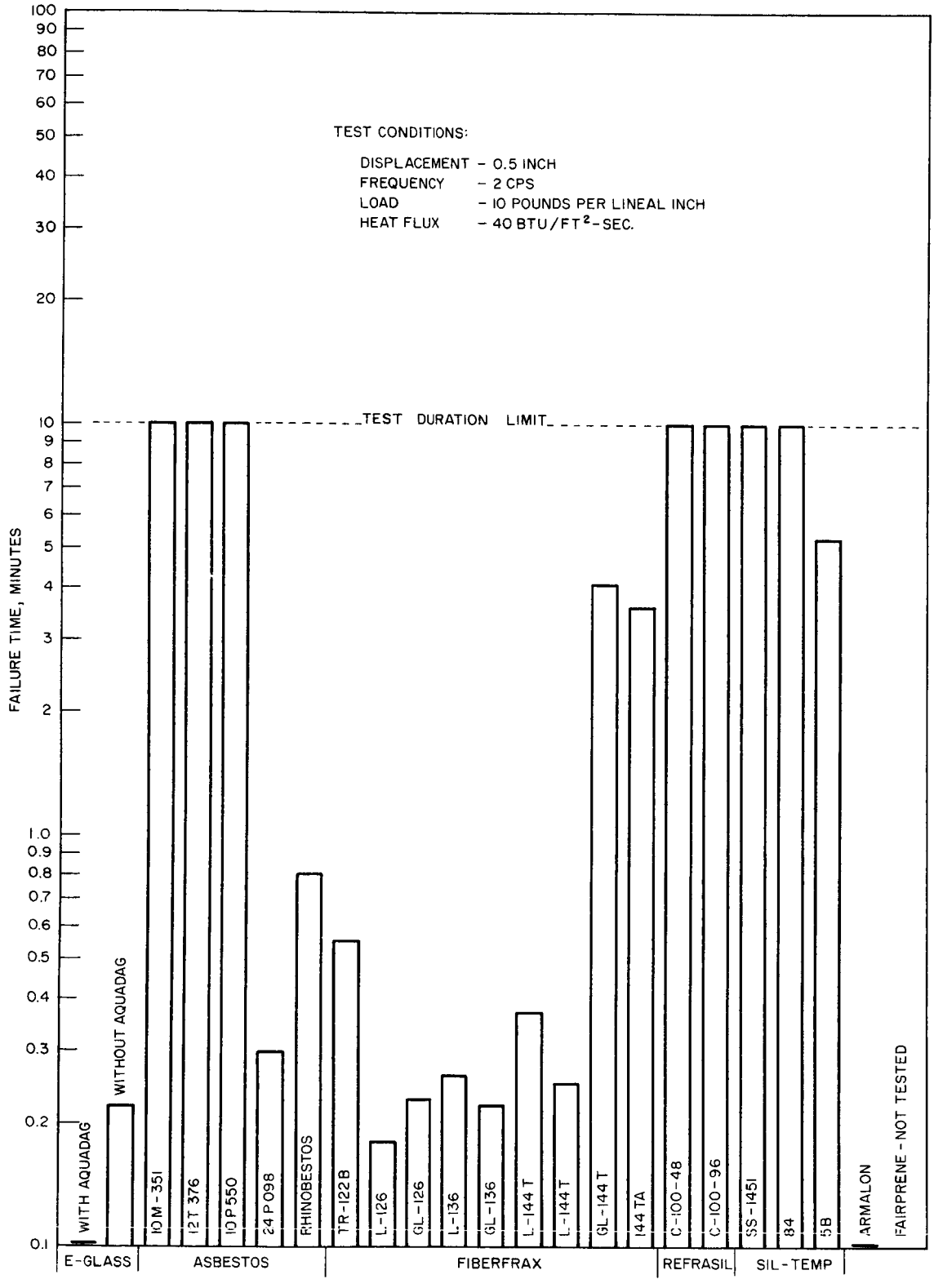


Figure 12. Ceramic fabric test results.

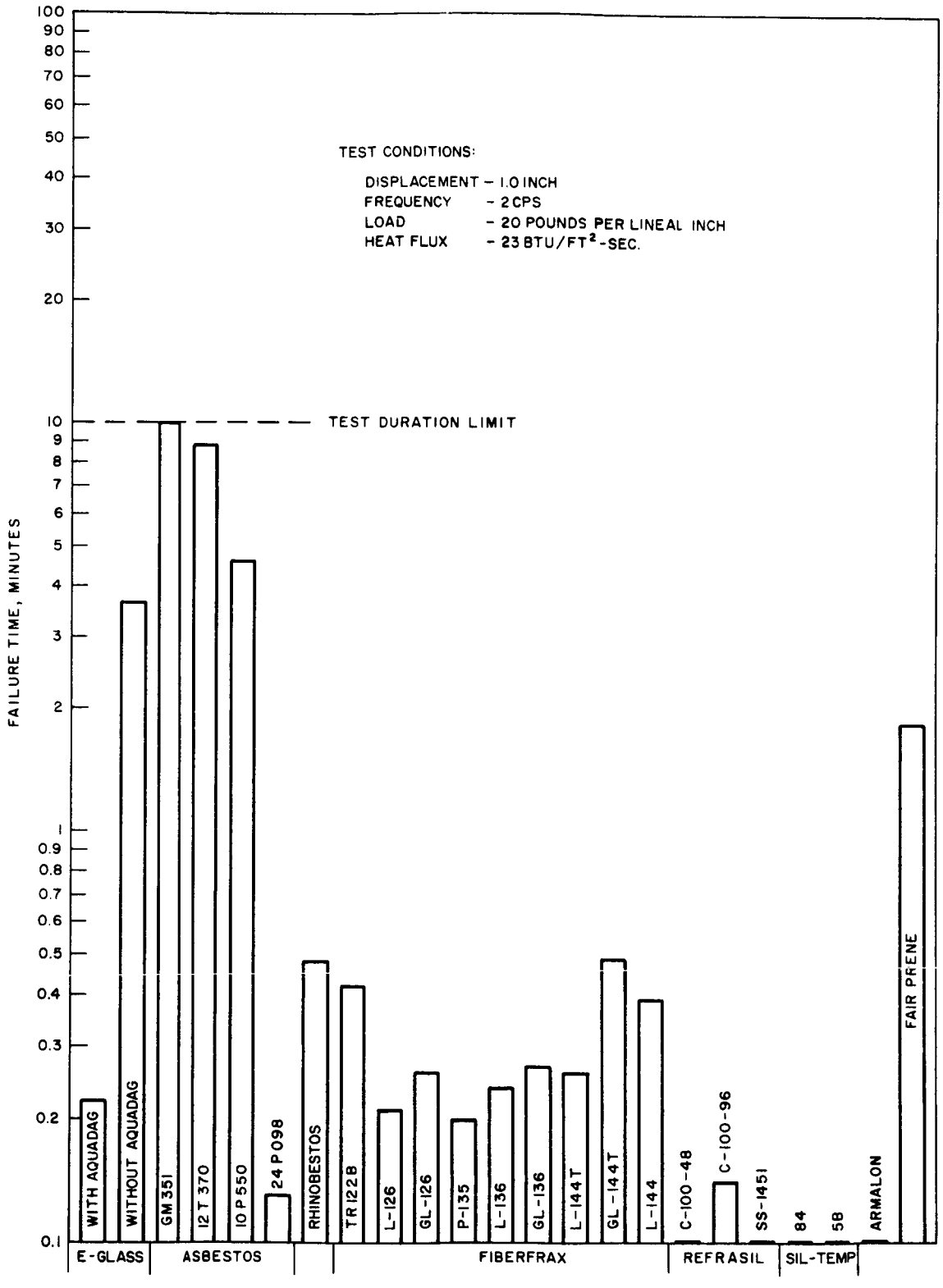


Figure 13. Ceramic fabric test results.

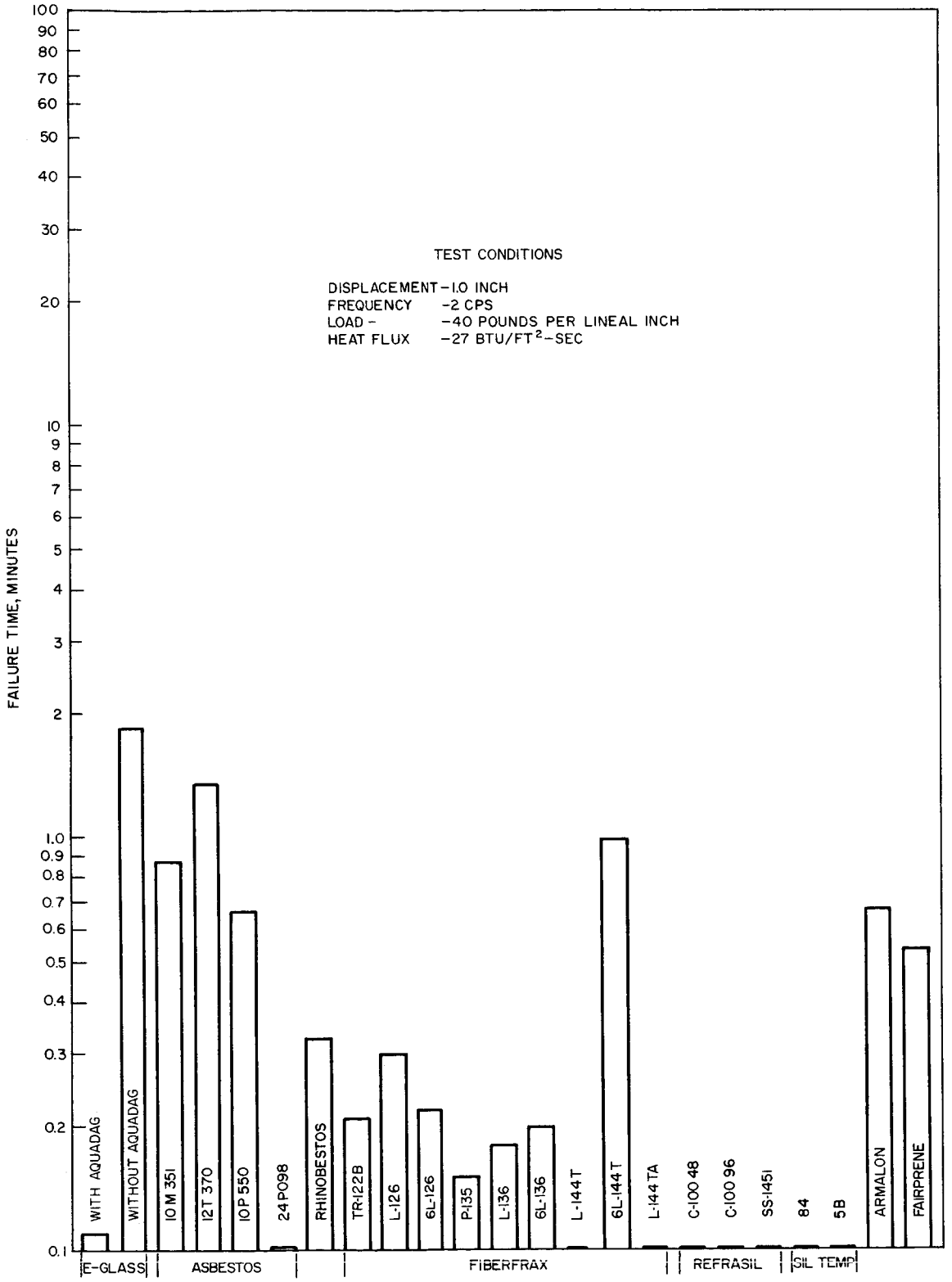


Figure 14. Ceramic fabric test results.

	Disp. Freq. Load. Heat Flux	0.5" 2 cps 10 lb 40 BTU	1.0" 2 cps 20 lb 23 BTU	1.0" 2 cps 40 lb 27 BTU
		Minutes to Failure		
E-Glass	181 w/Aquadag	0.02	0.22	0.12
	w/o Aquadag	0.22	3.67	1.83
Asbestos	10M351 AAAA	10.00	10.00	0.87
	12T370 AAAA	10.00	8.77	1.34
	10P550 AAA	9.60	4.58	0.66
	24P098 AA	0.30	0.13	0.00
	Rhinobestos	0.81	0.48	0.33
Fiberfrax	TR122B	0.55	0.42	0.21
	L126	0.18	0.21	0.30
	GL126	0.23	0.26	0.22
	P135	0.26	0.20	0.15
	L136	0.22	0.24	0.18
	GL136	0.37	0.27	0.20
	L144T	0.25	0.26	0.00
	GL144T	4.12	0.49	0.19
	L144TA	3.60	0.39	0.02
Refrasil	C-100-48	10.00	0.01	0.00
	C-100-96	10.00	0.14	0.00
	SS 1451	10.00	0.00	0.00
Sil-Temp	84	10.00	0.01	0.00
	5B	5.25	0.00	0.00
Armalon		0.01	0.05	0.67
Fairprene		not tested	1.86	0.54

Table 9. Results of fabric tests.

catastrophically showering fragments of burning rubber, while the front face supported the full load without further contribution from the rear face reinforcement. This material was not received in time for the first test, but it would have undoubtedly failed early under a heat flux of 40 BTU/ft^2-sec.

These tests indicate that a strong, high quality asbestos fabric faced with Refrasil or Sil-Temp for heat protection may provide the best combination of materials to resist the heat flux and loads anticipated in the flexible heat shield.

In order to determine the effects of reflective coatings on the life of fabrics subjected to the heat shield environment, Type 181 E-glass specimens were coated with inorganic pigments previously described. These specimens were subjected to similar dynamic fabric tests as shown with results in Figure 15. The magnesia coating improved the fabric lifetime by about fifty percent, suggesting that further work on reflective coatings may be very worthwhile.

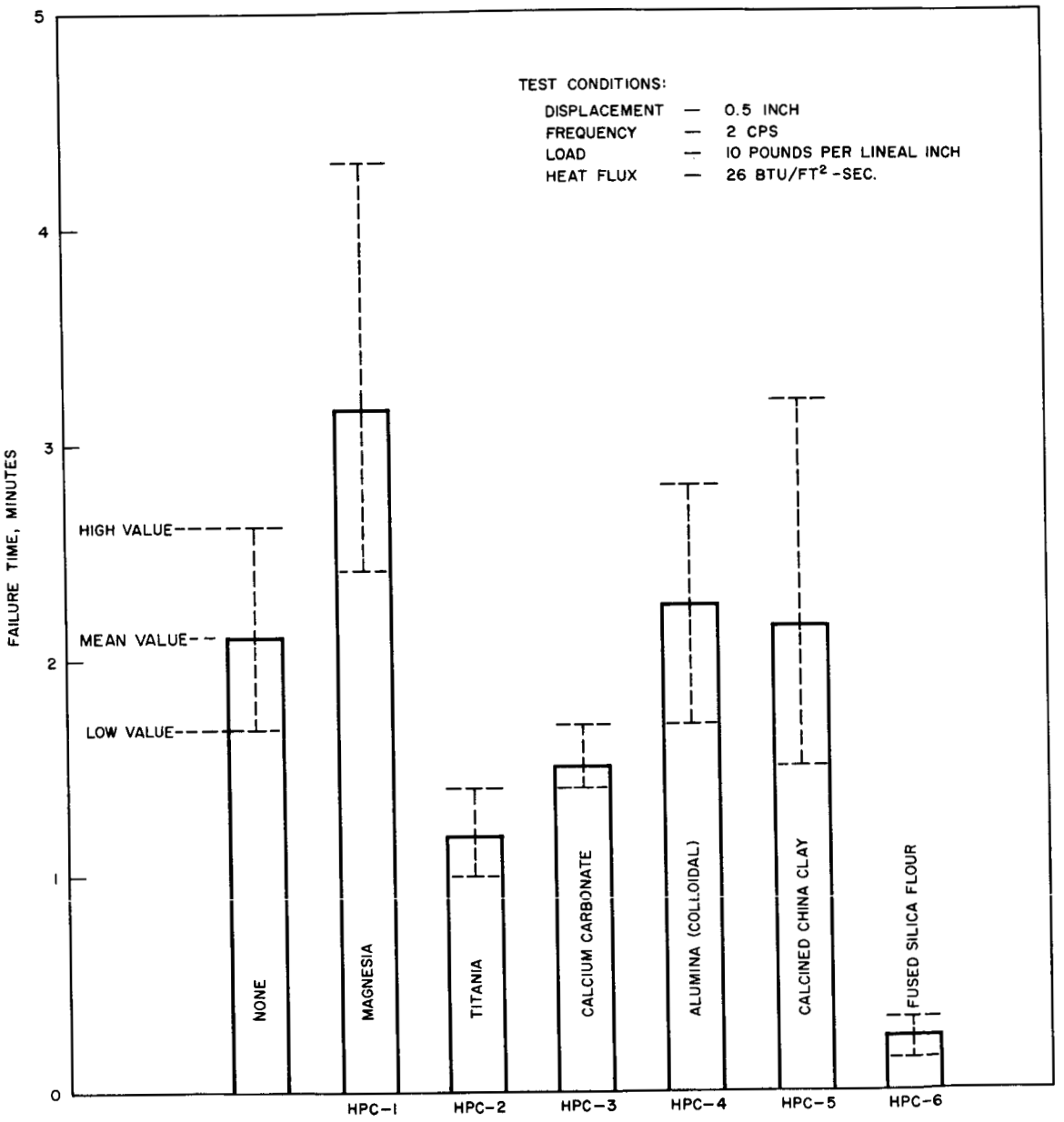


Figure 15. Test results of reflective coatings on Type 181 E-glass cloth.

IV. PERSONNEL

The personnel of the Ceramics Group, Advanced Techniques Section assigned to this project are listed below.

L. E. Gates	Member of the Technical Staff	Project Head
W. E. Lent	Associate Engineer	Fiberizing studies and fiber evaluation
A. A. Curme	Research Assistant	Fabric testing
C. J. Bahun	Member of the Technical Staff	Statistical analyses of fabric tests
N. H. Jensen	Laboratory Analyst	Fiber forming

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